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VOLUME 23 — December, 1946 — NUMBER 12

Magnetic Anistropy of Organic Solids, *P. W. Selwood and John Parodi*..... 574

The Chemistry of Tooth Tissues, *Ralph W. Phillips and Grant Van Huysen*..... 579

Catalysis and the Elementary Chemistry Course, *J. A. Campbell*..... 582

Selecting a Graduate School for Training in Chemistry, *John R. Sampey*..... 583

A Presentation of the Thermodynamic Functions, *C. C. Coffin*..... 584

Catalyzing the Approach to Equilibrium, *Joel H. Hildebrand*..... 589

Creative Chemistry Award..... 592

Photography as a Basic Science, *W. C. Oelke*..... 593

Recent Hypotheses Concerning the Mechanism of Photographic Development, *T. H. James*..... 595

A Versatile Microscope Hot Stage, *William A. Bonner*..... 601

Simple Devices for Measuring Out Small Quantities of Liquid, *F. C. Calkins*..... 604

Nonsilica Glass Systems, *F. L. Jones*..... 608

Instrumentation in Chemical Analysis, *Raymond H. Lambert*..... 610

Conversion of a Bomb-Release Control to Activate a Solenoid Stirrer, *Lynne L. Merritt, Jr., George F. Siddons, and John F. Suttle*..... 612

Division of Chemical Education of the American Chemical Society..... 618

Report of the New England Association of Chemistry Teachers

The Application of Colloid Chemistry to the Manufacture of Inflated Articles, *Langley W. Isom*..... 620

Annual Index

Author..... 627

Subject..... 629

Notice to Authors..... 636

Editor's Outlook..... 573

Letters..... 602

Out of the Editor's Basket..... 614

Recent Books..... 624

Frontispiece—World's Largest Bubble..... XXIII

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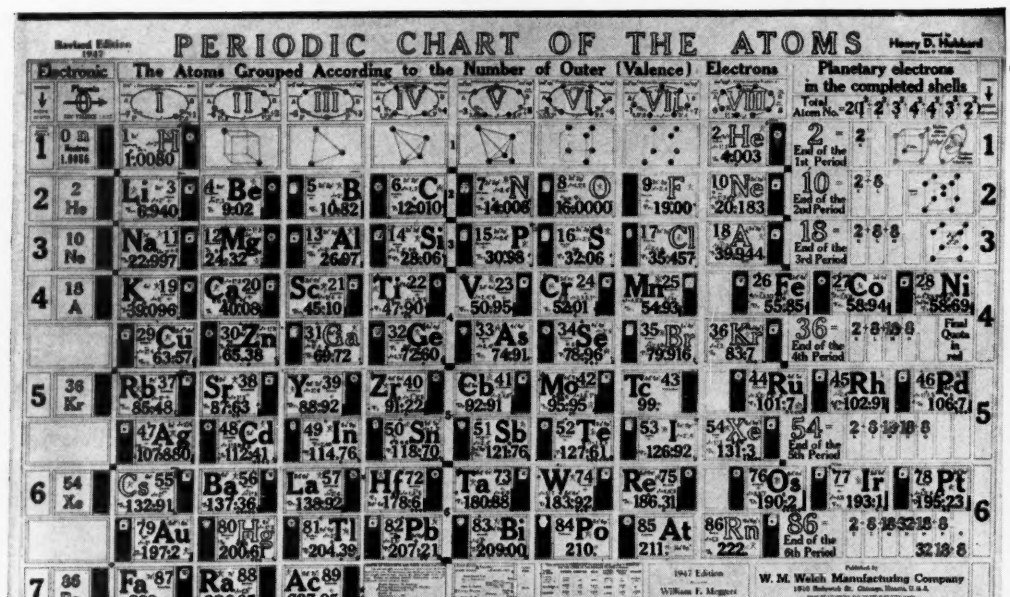

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Editor's Outlook

IN THESE days of upside-down economics, when one gets paid for work he doesn't do and has goods thrust upon him for which he doesn't pay, it is scarcely an event worth noticing when one is virtually pushed through the doors of opportunity on which he would not otherwise think to knock.

At first, many of us were inclined to think that the "G. I. Bill" with its more than generous educational advantages was as greasy a piece of pork as had ever been sliced off by the politicians. During the War the Army's order was: "Get out of college and into the Army"; the end of the War brought the slogan: "Join the Army and go to college." In either case college was a luxury, in the first instance to be dispensed with, in the latter to be used as attractive bait.

Nevertheless, it has worked out much better than many expected. A larger percentage than that predicted has taken advantage of the opportunity, has qualified for college, and is now enrolled—so large a percentage, in fact, that one rather wonders where the rest are. A visit to any of the nearest airports will partly answer that question. It seems as though almost every veteran who couldn't get into college has taken out his educational benefits by learning to fly in one of the hundreds of "aviation schools" that have sprung up, often consisting of little more than an airplane and a couple of flight instructors. Happy landings!

We haven't seen the final figures for the number of veterans who are now in our colleges and universities, but it certainly can best be expressed exponentially in powers of ten. Every higher education institution of which we have heard is literally bulging at the seams. But the expected lowering of standards doesn't seem to be taking place. These veterans, many of whom only qualified for entrance by the grace of God and the leniency of the Dean, are really making the grade—in fact, according to some reports, they are making all the *best* grades!

Now what does that show? That our American Way of Life is the best, that our educational system is tops, or just that our boys have what it takes? I'll settle for the last, although I won't give up the other two without a fight.

A boy fresh out of high school sometimes asks whether it wouldn't be better for him to take a couple of years "to get some experience" before going to college. It is seldom good advice to do so. The isolated individual generally finds that he loses the habit of studying, that his "experience" becomes a rut from which it is difficult to get out, and that the idea of going back to school among younger students doesn't appeal to him.

But the present case is different. There is the "group spirit" among these boys for one thing; there are so many of them, in fact, that in some cases they dominate the college picture. Their experiences have got them into no ruts, but in general have not only matured them but also given them increased appetite for more education and training.

The whole program will cost you and me and the rest of the country a great deal of money, but it is well invested—better than in subsidizing veterans in the purchase of houses, businesses, or automobiles.

WE OCCASIONALLY hear complaint that the scientist did not get his just deserts in the late war but was kicked around a good bit by "the military." It is beginning to appear that in this country he fared much better, in his efforts to be useful, than did his counterpart in some of the enemy countries. Hitler was notoriously contemptuous of science and scientists and reaped the rich harvest of this oversight. The Japanese scientist was also an "underdog." One of them described his experiences in a recent report. He and some of his colleagues among the meteorologists and oceanographers conceived the very reasonable and helpful plan of making use of the ocean currents to *float* supplies of sugar, gasoline, etc., from Formosa and Manchuria to the Japanese islands, when the American blockade made shipping precarious.

> On a snowy day in February, 1945, I, accompanied by Drs. Fujiwhara and Sekiguti, recommended this plan to the Naval and Military Technical staffs. The reason why we recommended this plan again and again, and to different personnel, was that they were so inactive in adopting plans recommended by scientists. This, I suppose, was partly because the military men did not trust scientists and because the Japanese people in general were placed on a low scientific level. . . .
>
> Meanwhile Dr. R. Kinoshita, Professor in Pathology, Osaka Imperial University, told me that most Japanese people were now losing their efficiency as the effect of hunger edema resulting from the lack of amino acids. He said if the present condition of public nourishment persisted until the end of the year, the health of the Japanese people would so decline that Japan would lose its working power. It was a serious question how to prevent so dangerous a state. Kinoshita insisted that the only remedy for this was to import soybeans from Manchuria, utilizing the currents in the Japan Sea. Based on these circumstances, Kinoshita and I explained during March the importance of the transport of soybeans from Manchuria to various personnel in Government, Army, and Navy. But there was no reaction.[1]

[1] *Trans. Am. Geophys. Union*, **27**, 519 (1946).

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MAGNETIC ANISOTROPY of Organic Solids

P. W. SELWOOD and JOHN PARODI
Northwestern University, Evanston, Illinois

INTRODUCTION

MAGNETIC anisotropy is the effect wherein a substance shows different magnetic properties along different directions in space. The effect was discovered by Plücker about the year 1848, and it was extensively investigated by Michael Faraday. The application to structural problems in organic chemistry dates from 1927, but the method seems to be practically unknown in the United States. This review is based almost entirely on the papers of Krishnan and of Mrs. Lonsdale.

THEORY OF DIAMAGNETISM

In diamagnetic substances the magnetic flux density is less than in the surrounding field, and the susceptibility is independent of temperature. With few exceptions organic compounds are diamagnetic. The classical theory of diamagnetism is due to Langevin.

An electron of mass, m, moving in a closed orbit is equivalent to a current in a wire. The magnitude of this equivalent current is

$$\frac{\epsilon}{c} \cdot \frac{\omega}{2\pi}$$

electromagnetic units, where ϵ is the electronic charge,

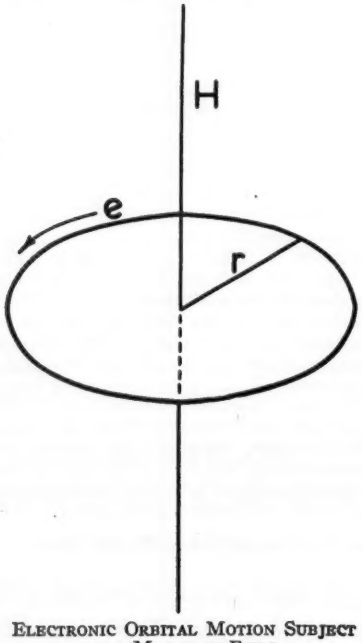

FIGURE 1. ELECTRONIC ORBITAL MOTION SUBJECT TO A VERTICAL MAGNETIC FIELD

c is the velocity of light, and ω the angular velocity of the electron. The magnetic field produced by this current will be of the same intensity at a given distance as a magnet of moment μ, such that

$$\mu = \frac{\epsilon\omega S}{2\pi c} = \frac{\epsilon\omega\pi r_1^2}{2\pi c} = \frac{\epsilon\omega r_1^2}{2c}$$

where S is the area of the bounded surface around which the electron moves, and r_1 is the radius of the orbit. If the orbit is elliptical, ωr_1^2 may be replaced by an average so that

$$\mu = \frac{\epsilon\overline{\omega r_1^2}}{2c}$$

Larmor has shown that the effect of a perpendicular field H on such a system superposes on the motion of the electron an angular velocity of precession given by the expression

$$l = -\frac{\epsilon H}{2mc}$$

The magnetic moment associated with the orbit will undergo a change,

$$\Delta\mu = \frac{\overline{\epsilon r_1^2(\omega + l)}}{2c} - \frac{\overline{\epsilon r_1^2\omega}}{2c}$$

$$= \frac{\overline{\epsilon r_1^2 l}}{2c}$$

$$= -\frac{\epsilon^2\overline{r_1^2}H}{4mc^2}$$

For a system of n orbits oriented at random to the field, the mean square radius of the projected orbit perpendicular to H becomes

$$\overline{r_1^2} = \overline{2/3 r^2}$$

where $\overline{r^2}$ is the mean square radius of the orbit. For each gram atom the change of magnetic moment will be

$$\Delta\mu_a = -N\frac{\epsilon^2 H}{6mc^2}\sum_n \overline{r^2}$$

where N is Avogadro's number. The magnetic susceptibility per gram atom is then

$$\chi_a = \frac{\Delta\mu_a}{H} = -N\frac{\epsilon^2}{6mc^2}\sum\overline{r^2} = -2.83\sum\overline{r^2}\times 10^{10}$$

Diamagnetism depends only on the radius of the electronic orbits and is therefore independent of temperature. Quantum mechanics gives the same expression for χ_a.

MAGNETIC ANISOTROPY

A substance which is truly isotropic is one in which the atoms are all arranged at random, as for instance in a gas, most liquids, and amorphous solids. A substance may be effectively isotropic if it is composed of microcrystals of completely random orientation.

In crystals of low symmetry there are three mutually perpendicular directions known as the axes of principal magnetization along which the direction of magnetization coincides with the direction of the applied field. The magnetic susceptibilities along these axes are known as "principal" susceptibilities, and their values are often unequal.

A piece of noncubic crystalline matter suspended in a nonuniform field will tend to orient itself so that the axis of maximum susceptibility in the plane of rotation lies along the lines of force. If the substance is diamagnetic, this will be the axis of minimum numerical susceptibility.

The magnetic anisotropy is generally expressed as the difference of two principal susceptibilities. Uniaxial crystals have only two principal susceptibilities, along and perpendicular to the principal crystal axis. Crystals such as these include many metals, some of the common minerals, and natural and synthetic fibers.

If a substance is powdered, the average susceptibility of the powder is found to be the mean of the three principal susceptibilities, or

$$\bar{\chi} = \frac{1}{3}(\chi_1 + \chi_2 + \chi_3)$$

In orthogonal crystals the axes of principal susceptibility coincide with the crystal axes. In the monoclinic system one principal axis coincides with the symmetry axis of the crystal. Many organic compounds crystallize in the monoclinic system.

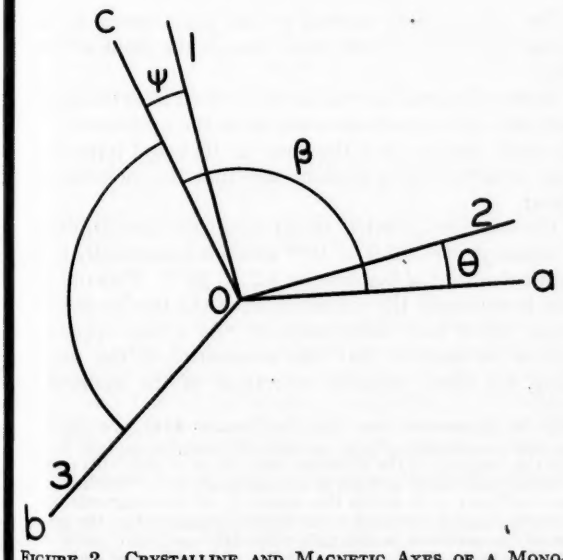

FIGURE 2. CRYSTALLINE AND MAGNETIC AXES OF A MONOCLINIC CRYSTAL, *a*, *b*, *c* AND *1*, *2*, *3*, RESPECTIVELY

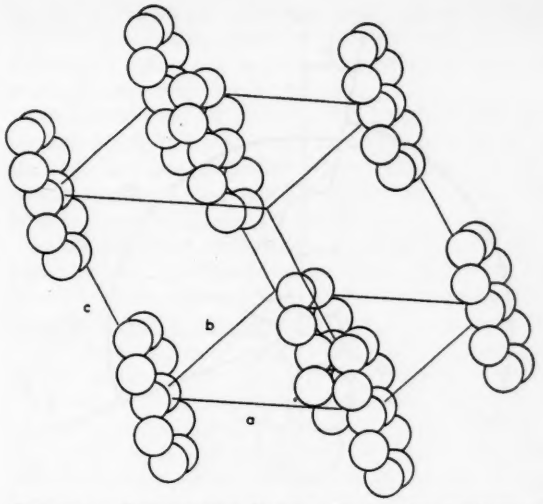

FIGURE 3. NAPHTHALENE CRYSTAL CELL. TWO MOLECULES AT EITHER END OF THE UNIT CELL DIAGONAL ARE OMITTED SO THAT THE MOLECULES IN THE CENTER OF THE TOP AND BOTTOM FACES MAY BE SEEN

ANISOTROPY OF ORGANIC COMPOUNDS

The average (powder) susceptibility of organic compounds is additive, like molar refractivity; hence, even in the solid state the mutual influence of neighboring molecules on the magnetic susceptibility must be negligible. The anisotropy of a single crystal must be due to the anisotropy of the unit cell, and that in turn depends only on the resultant anisotropy of the individual molecules in the cell. If the electron distribution in the molecule is such that the molecule is essentially isotropic from the magnetic standpoint, then the crystal will show little or no anisotropy. If the molecule is magnetically anisotropic, then the degree to which the crystal reveals that anisotropy depends entirely on the arrangement of the molecules. If the molecules are arranged in more or less parallel positions, as in layer or chain structures, then the crystal anisotropy will be nearly as large as that of the molecule, or, the molecules may be arranged so that their anisotropies cancel out as, for example, in cubic crystals.

The largest diamagnetic anisotropies are shown by aromatic compounds, the molecules of which contain benzene, cyanuric, or phthalocyanine rings. Many such substances have undergone complete structure determinations by X-ray methods and the orientations of the molecules in the crystal are known. It is therefore possible to deduce from the principal susceptibilities and the molecular direction cosines the principal susceptibilities of the individual molecules. A few examples are given in the following table:

PRINCIPAL MOLAR SUSCEPTIBILITIES (K) OF INDIVIDUAL MOLECULES

Compound	$-K_1 \times 10^6$	$-K_2 \times 10^6$	$-K_3 \times 10^6$
Benzene, C_6H_6	37.4	37.3	91.2
Naphthalene, $C_{10}H_8$	39.4	43.0	187.2
Anthracene, $C_{14}H_{10}$	45.9	52.7	272.5
Metal-free phthalocyanine, $C_{32}N_8H_{18}$	165	120	982

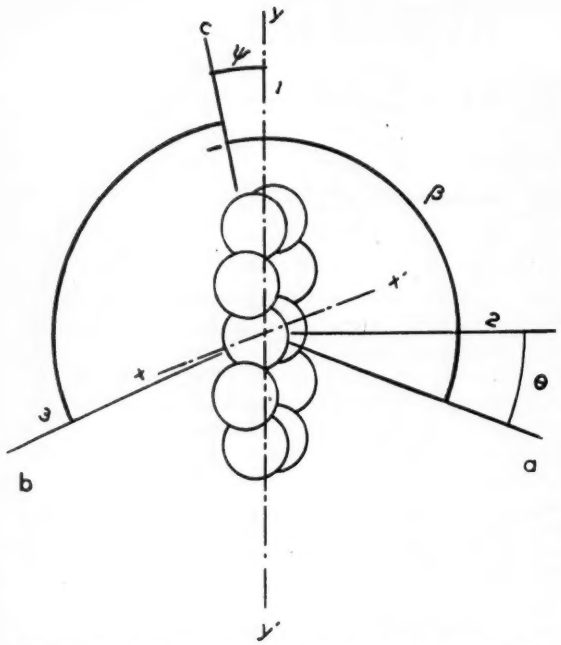

FIGURE 4. A NAPHTHALENE MOLECULE IN RELATION TO ITS MAGNETIC AND CRYSTALLINE AXES. THE MUTUALLY PERPENDICULAR AXES, XX' AND YY', FIX THE POSITION OF THE PLANE OF THE MOLECULE, YY' BEING THE LONG AXIS OF THE MOLECULE

X-ray analysis shows that all these molecules possess a plane configuration: K_1, K_2 lie in the plane of the molecule, K_3 is perpendicular to the plane.

Various suggestions have been made to explain the abnormal diamagnetism along K_3. The most popular explanation is that the aromatic, or resonance, electrons occupy orbits of molecular instead of atomic size.

In favorable cases it is possible to estimate the anisotropy of the individual molecules with some accuracy. It is then possible to deduce the orientations of these molecules from magnetic measurements on the single crystals. The magnetic method thus may provide valuable aid in the earlier stages of an X-ray investigation or occasionally may suffice for molecular orientation studies without the aid of X-rays.

Carbon crystallizes in two forms, diamond (cubic) and graphite (hexagonal). Diamond is isotropic, but graphite is strongly anisotropic. The principal molar susceptibilities along and normal to the hexagonal axis are

$$\chi_{\parallel} = -264 \times 10^{-6} \text{ and } \chi_{\perp} = -6 \times 10^{-6}$$

$\chi_{\parallel}$ is strongly dependent on temperature, particle size, and the presence of impurities. The usual explanation is that there are loosely bound electrons occupying large orbits in the plane of the rings.

Naphthalene belongs to the monoclinic class of crystals. Figure 2 depicts the crystalline and magnetic axes of a monoclinic crystal. Two of the principal magnetic axes lie in the (010) or ac plane; the magnetic susceptibilities along these axes are χ_1 and χ_2, χ_1 being greater than χ_2. The angle, ψ, which the χ_1 axis makes with the c axis of the crystal, taken as positive toward the obtuse angle, β, between the c and a axes, determines the positions of the two magnetic axes. The angle, θ, is connected with ψ by the relation, $90° + \theta + \psi =$ obtuse β. The third magnetic axis is along the b axis and the molar susceptibility along it is χ_3.

X-ray studies show that the crystal unit cell of naphthalene contains two molecules, for each of which the structure is plane. Both molecules have their lengths, the lines which join centers of constituent rings, in the (010) plane, making an angle of 12° with the c axis.[1] Considerations of symmetry show that the long axis of the naphthalene molecule must be one of its magnetic axes. We should, therefore, expect the above direction in the crystal to be one of the magnetic axes of the crystal. Direct observation of the magnetic axes confirms this view, and the angle ψ is actually found to be 12°. The other two magnetic axes are along the breadth of the molecule in the plane of the rings and along the normal to the plane of the rings, respectively.

X-ray studies also show that the planes of the molecules are inclined at +65° and −65°, respectively, to the (010) plane. We have, therefore, all the information necessary to find the magnetic susceptibilities of the individual molecule. We have

$$\chi_1 = K_1$$
$$\chi_2 = K_2 \cos^2 65° + K_3 \sin^2 65°$$
$$\chi_3 = K_2 \sin^2 65° + K_3 \cos^2 65°$$

where χ and K are principal molar susceptibilities of crystal and molecule, respectively.

Substituting the experimentally determined values for χ_1, χ_2, χ_3, we have

$$K_1 = -39.4 \times 10^{-6}$$
$$K_2 = -43.0 \times 10^{-6}$$
$$K_3 = -187.2 \times 10^{-6}$$

The susceptibility normal to the plane seems to be almost four and one-half times that in the plane of the ring.

Biphenyl's structure will be determined from the magnetic data instead of vice versa as in the previous case. We shall assume that the rings in biphenyl have the same structure as in benzene and that the molecule is planar.

The average (powder) molar magnetic susceptibility of biphenyl is -102.9×10^{-6} which is numerically less than twice that of benzene by 7.7×10^{-6}. This difference is obviously the contribution of the two hydrogen atoms which have been dropped. As a first approximation we assume that this diminution is the same along the three principal directions of the molecule.

[1] J. M. Robertson [*Proc. Roy. Soc. London*, **A142**, 674 (1933)] has made a complete X-ray analysis of naphthalene and finds that the long axis of the molecule does not lie in the (010) plane of the crystal, but is inclined at a small angle to it. With reference to Figure 4 in which the origin, O, of the magnetic and crystalline axes is not labeled, the following angles place the position of the molecule in the unit cell: $bOY = 102.6°$, $aOY = 115.3°$, $bOX = 28.8°$, $aOX = 71.2°$. Dimensions of the unit cell: $a = 8.29$ A.. $b = 5.97$ A., $c = 8.68$ A., $\beta = 122.7°$.

The principal susceptibilities for the benzene molecule are $K_1 = -37.3$, $K_2 = -37.3$, and $K_3 = -91.2$ ($\times 10^{-6}$). We then obtain for the principal molar susceptibilities of the biphenyl molecule

$$K_1 = K_2 = -(2 \times 37.3) - 7.7 = -66.9 \ (\times 10^{-6})$$
$$K_3 = -(2 \times 91.2) - 7.7 = -174.7 \ (\times 10^{-6})$$

On the other hand, the principal molar susceptibilities for the biphenyl crystal are

$$\chi_1 = -63.4 \times 10^{-6}$$
$$\chi_2 = -146.5 \times 10^{-6}$$
$$\chi_3 = -98.9 \times 10^{-6}$$

and the angle $\psi = 20.1°$.

A correlation of these two sets of constants gives orientations for the two molecules in the unit cell of biphenyl as follows.

Place both molecules with their planes parallel to (100) and their lengths along the c axis. Now, to bring the molecules to their actual orientations in the crystal they are given the following rotations:

First, a rotation about the c axis of one of the molecules through an angle, λ, and of the other through an angle, $-\lambda$.

Second, a rotation of both molecules about the b axis through an angle, ζ, the positive direction of the rotation being defined as being from the c axis to the a axis, through the obtuse angle β.

Third, a rotation of the molecules through angles $+\nu$ and $-\nu$, respectively, about the normal to the plane which contains the b axis and the direction of lengths of the molecules after the second rotation has been performed.

It is clear that $\zeta = \psi = +20.1°$ and λ and ν can be obtained from the following relations:

$$\chi_1 = K_1 \cos^2 \nu + (K_2 \cos^2 \lambda + K_3 \sin^2 \lambda) \sin^2 \nu$$
$$\chi_2 = K_2 \sin^2 \lambda + K_3 \cos^2 \lambda$$
$$\chi_3 = K_1 \sin^2 \nu + (K_2 \cos^2 \lambda + K_3 \sin^2 \lambda) \cos^2 \nu$$

We have already tacitly assumed that $\chi_1 + \chi_2 + \chi_3 = K_1 + K_2 + K_3$; therefore, only two of the above relationships are independent. Solving, we get

$$\lambda = 31°, \quad \nu = 0°$$

We find that the lengths of the molecules lie in the (010) plane in the obtuse angle β at 20.1° to the c axis, while the planes of the molecules are inclined at plus and minus 31°, respectively, to the b axis.[2]

EXPERIMENTAL METHODS

The obvious approach to anisotropy measurements is to use the familiar Gouy or Faraday magnetic susceptibility balances but to orient the single crystal so that only one magnetic axis is subjected to the field in the line of motion. The force exerted on the sample may be measured directly, or the sample may be surrounded with a solution of such susceptibility that no motion of the sample occurs on application of the field. This method suffers from the disadvantage that it is difficult to orient and mount the samples so that the principal axes are properly directed.

An isotropic substance will suffer no orientation in a nonuniform field if the sample is cut to spherical or, as the case may be, cylindrical shape. Alternatively, the sample may be mounted in a uniform field. This is the basis of a method devised by Krishnan.

In the Krishnan method the sample is suspended by a torsion fiber in a homogeneous field. A field of sufficient homogeneity may be found in a small region between relatively large plane pole pieces. In general the crystal may be placed so that one magnetic axis is in the axis of the torsion fiber. If the crystal is isotropic, it will not move on application of the field. If the crystal is anisotropic, it will turn against the torsion of the fiber until the largest (smallest diamagnetic) axis approaches the direction of the lines of force. The torsion head may now be turned until the crystal suffers no orientation on application of the field. The largest numerical susceptibility is now parallel to the field. During this orientation operation the angle ψ may be found by visual observation, provided the b axis has been mounted vertical.

If now the crystal is made to oscillate, the period of oscillation is related to the molar magnetic anisotropy as follows

$$\chi_1 - \chi_2 = \frac{t_1^2 - t^2}{t^2} \cdot \frac{C}{H^2} \cdot \frac{M}{m}$$

where t and t_1 are the periods of oscillation with the field on and off, respectively, C is the torsional constant of

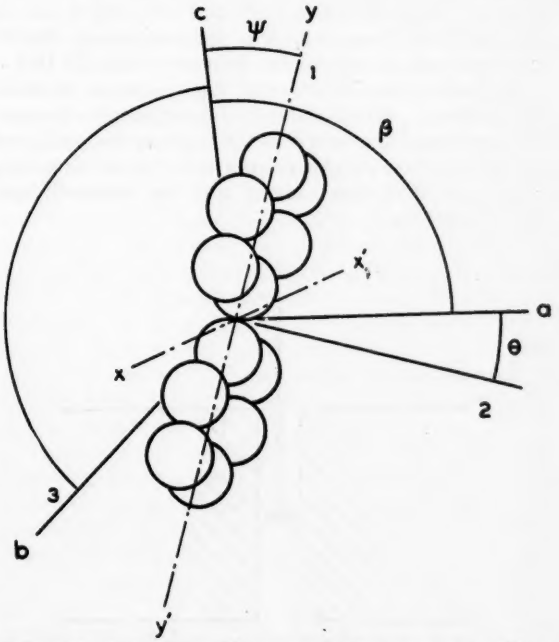

FIGURE 5. A BIPHENYL MOLECULE IN RELATION TO ITS MAGNETIC AND CRYSTALLINE AXES

[2] Independent X-ray analysis by J. Dhar [*Ind. J. Phys.*, 7, 43 (1932)] confirms the work of Krishnan. Dhar determined: $\psi = 20°$, $\lambda = 32°$, and $\nu = 0°$. Dimensions of the unit cell are: $a = 8.22$ A., $b = 5.69$ A., $c = 9.5$ A., and $\beta = 94.8°$.

the fiber, m is the mass of the crystal, M is the molecular weight, and H is the field. The anisotropy observed is, of course, that existing in the plane of oscillation. The third principal susceptibility may be investigated by a different crystal orientation. If absolute values are required, one of the principal susceptibilities must be measured by one of the direct methods; or, alternatively, the average (powder) susceptibility may be found, although this is somewhat less accurate.

By this elegant method Krishnan and his coworkers have studied crystals weighing about 0.1 mg. The method has obvious applications to solids in which the crystal axes may be poorly defined, as in synthetic polymers. The method could also be extended quite easily for measurements over a wide range of temperature, although little is known concerning temperature dependence of the anisotropy of organic solids.

ANISOTROPY OF HIGH POLYMERS

Very little work has been done on the magnetic anisotropy of polymeric material, but the method would appear to have some useful applications.

Mme. Cotton-Feytis has recently demonstrated the anisotropy of several natural types of fibers and of crude rubber. These substances may, to a first approximation, be considered as uniaxial crystals. The largest numerical susceptibility is sometimes longitudinal, sometimes perpendicular, to the long axis. Various types of cellulose (cotton, sisal, etc.), silk fiber, keratin, and collagen, all show varying degrees of anisotropy. The magnitude of the anisotropy roughly parallels the degree of molecular orientation as revealed by X-ray studies. For example, a normal sample of collagen showing a high degree of molecular orientation has an anisotropy ten times as large as the same sample shrunk and rendered amorphous by treatment with HCHO.

Nilakantan has investigated the anisotropy of wood and cellulose. Wood is anisotropic, apparently because of a molecular anisotropy in the cellulose molecule and the more or less regular orientation of these molecules along the fiber axis. Lignin and the hemicelluloses are amorphous.

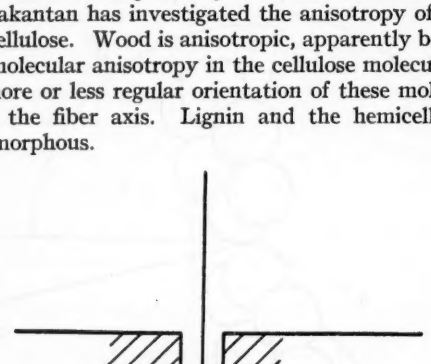

FIGURE 6. THE KRISHNAN MAGNETIC TORSION BALANCE. THE SAMPLE IS SUSPENDED FROM A THIN QUARTZ FIBER IN A UNIFORM MAGNETIC FIELD BETWEEN FLAT POLE PIECES

The greatest diamagnetism of cellulose is along the fiber axis, which is presumably along the length of the molecule. This anisotropy in α-cellulose derived from teakwood is about $\chi_{\parallel} - \chi_{\perp} = -0.1 \times 10^{-6}$. The average susceptibility of α-cellulose in powder form is -0.508×10^{-6}. The method appears to have some utility in confirming the molecular orientations of cellulose shown by X-ray studies.

Crude rubber normally has a certain degree of anisotropy which is altered by hot and cold working and by compression and tension. Plexiglass is said by Cotton-Feytis to yield somewhat similar results, but no details are given.

When crude rubber is stretched, the anisotropy increases but seems to tend toward a limit. This parallels the effects observed with X-ray methods and suggests more or less complete molecular orientation in one direction at high elongations. The anisotropy observed in stretched rubber is, incidentally, quite large. The figures reported by Cotton-Feytis for oscillation periods with the magnetic field off and on, respectively, are 85 seconds and 6.2 seconds. There is no doubt that the method is a sensitive one for detecting molecular orientation in such systems.

The effects observed will doubtless be much more valuable in the study of high polymers containing aromatic groups. It will be recalled that normal to the plane of the rings the principal susceptibility is very large for aromatic compounds. It seems probable that degrees of molecular orientation quite beyond detection by other methods will be readily estimated by the anisotropy measurements. This applies both to gross orientation of the molecule and to side chains, particularly if these are aromatic. There are obvious applications of such studies to changes occurring under compression, tension, extrusion, hot and cold working, and aging. The establishment of relationships between anisotropy and physical properties, is also a possibility.

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The Chemistry of Tooth Tissues

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TOOTH decay is such a prevalent disease that it affects 95 per cent of the individuals in this country. While it is true that many adults live apparently healthy lives without natural teeth, there is always the danger that dental disease, unchecked, may result in generalized infection and severe illness. Then, too, replacement of tooth tissues that have been lost and destroyed by tooth decay with fillings, bridges, and plates is an expensive and time-consuming procedure.

The belief that refined carbohydrates, such as sugar, exert a harmful effect on teeth has been taught by the dental profession and accepted by the public for many years. This idea has persisted in spite of the fact that, biochemically, very little is actually known about the oral cavity or what happens to the various types of ingested carbohydrates in the mixed saliva which bathes the teeth. Actually, the causes of tooth decay are still a long way beyond our understanding. For that matter, we have just begun to do intelligent research.

We do know that tooth decay is a surface phenomenon and that during the early phases before the cavity is formed, there is a change in the tooth surface equilibrium. Bacteria grow on practically all tooth surfaces constantly and harmlessly. However, in certain protected regions, apparently the physiochemical equilibrium becomes disturbed sometimes so that these organisms cease to be harmless and tooth surface destruction takes place.

To bring about the formation of a cavity, some change in the enamel surface must occur. As yet, we know too little about the morphology of the surface and its physiochemical properties. There are two ways, of course, in which a surface may become changed. It may be the result of either mechanical abrasion or molecular exchange. Both of these conditions prevail upon the teeth. On the one hand, one may find shallow cavities formed at the necks of some teeth. These cavities have smooth polished surfaces and for a long time were thought to be due to an erosion by an acid secreted by the adjacent soft tissue. It has now been shown that these cavities are caused by abrasions brought about primarily by improper use of the tooth brush plus perhaps an abrasive dentifrice. On the other hand, there can be no doubt that the other more common lesion—that is, tooth decay—starts as a disturbance or early molecular surface change. Before either process can be studied, it is of course necessary that more is learned about the conditions of normal tooth surfaces. This is true both from a morphological and a physiochemical standpoint.

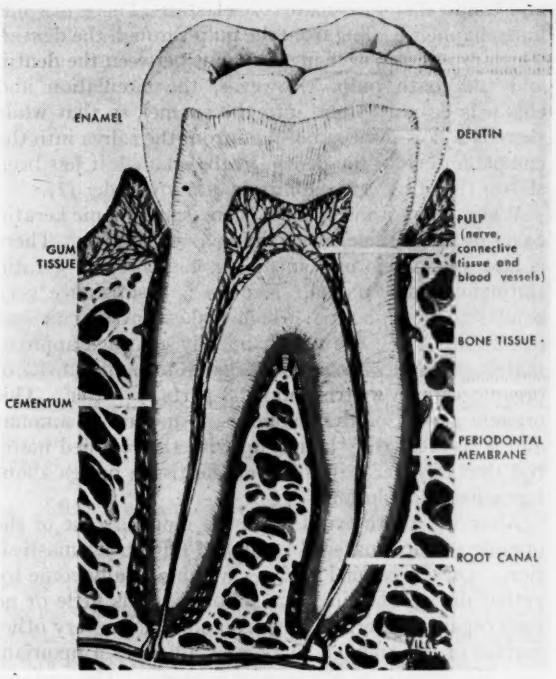

FIGURE 1.—TOOTH AND SUPPORTING STRUCTURES

ENAMEL—MICROSCOPIC APPEARANCE

The enamel of the tooth is a very hard structure covering that part which extends above the gum tissue. This is known as the tooth crown. The enamel covering varies from a thickness of about 2 mm. on the biting edge to the thinness of a razor-blade at the neck of the tooth. This enamel is composed of microscopic rods about 0.005 mm. in diameter, and the length of each rod extends throughout the thickness of the enamel covering. The rods are so closely cemented to each other along their sides that it is impossible to separate them. Their general direction is radial, but they describe many curves and crisscrosses throughout their length. This enamel is the hardest tissue of the body, and as a covering of the tooth it is the part which usually comes in contact with the saliva, mouth debris, and myriads of bacteria in the saliva.

The enamel is so hard and brittle that it would fracture even under ordinary biting stresses unless it were supported by the elastic bone-like dentin which, as one can readily see in Figure 1, forms the bulk of the tooth, crown, and root. This dentin, although hard like bone, is not so hard nor nearly so brittle as the enamel which it supports. The rest of the tooth is composed of a cavity containing blood vessels, nerves, and other soft tissues. This is called the pulp. The soft tissue pulp and its blood circulation keep the dentin relatively

moist, and therefore relatively elastic. There are minute channels leading from the pulp through the dentin, which provide an exchange of fluids between the dentin and the tooth pulp. However, the circulation and channels do not extend into the enamel, so that while there is a slow diffusion of fluid from the saliva into the enamel and from the dentin to the enamel, it has been shown that this permeability is of a low order (*1*).

When the tooth is formed, there is an organic keratin covering, or cuticle, over the enamel surface. There is also an organic protein matrix composed of keratin throughout the enamel. Keratin is a substance very similar to that material which makes up the hair and fingernails. The enamel chemically contains approximately 96 parts of inorganic calciferous material, 1.7 of organic keratin matrix, and 2.3 parts of water. This organic matrix of the enamel is so minute in amount and is so intimately bound up with the calcified material that any decalcification of the tissue brings about immediate dissolution.

After the tooth erupts into the mouth, some of the organic surface material is rubbed off during mastication. On the enamel surfaces which actually come together during chewing, one probably finds little or no such organic material; however, on almost every other surface of the enamel, there is at all times a luxuriant mat-like growth of filamentous bacteria. The surfaces which are kept clean by the contact of teeth during mastication very seldom show tooth decay. The greater surfaces of the enamel which are exposed to the excursion of food do appear under normal conditions bright and polished. A microscopic examination of these surfaces, however, shows the presence of many bacteria. These bacteria apparently thrive without damage to the enamel. However, in certain protected areas, such as the pits and fissures and the surfaces in between teeth, one will find all too frequently areas in which the by-products of bacterial growth are presumably decalcifying tooth surfaces. It is on these protected surfaces that chemical change of the enamel itself is gradually brought about and cavities eventually develop. It is the chemical composition of this enamel surface that must be studied so that the changes associated with the decalcification of tooth decay may be understood.

THE CHEMISTRY OF ENAMEL

In order to further the knowledge of dental caries, chemists and dentists have for many years been attempting to determine the chemical constitution of enamel and dentin. As yet, the molecular construction is not definitely known. Research, by X-ray diffraction in particular (*2*), has shown a marked similarity between certain naturally occuring apatites and tooth substances. This work, and the chemical studies which determined the Ca:P ratio in enamel to be 2:15, seems to indicate that the main molecular constituent of enamel is a complex salt, mainly $Ca_3(PO_4)_2 \cdot Ca(OH)_2$. The compound is called hydroxy-apatite. This calcium compound can be found in nature as can various other metallic hydroxy-apatites. Some $CaCO_3$ is apparently also present in enamel and is usually found as the compound $Ca_3(PO_4)_2 \cdot CaCO_3$. Various other unexpected elements are present in small quantities, such as 0.05 per cent Cl, 0.02 per cent Pb, and 0.02 per cent Fe.

Although the inorganic chemical composition of enamel is similar to dentin, there is a much greater amount of organic material in dentin. This results in a marked difference in hardness between the two tissues. Figure 2 shows Knoop indentations on a polished surface of enamel and dentin at the dentino-enamel junction. These indentations are made by means of a diamond point under constant load. The length of the indentations are indicative of the corresponding hardness for the enamel and dentin; the longer the indentation, the softer the material. As shown in this picture, the indentation in the dentin is much longer than the one made in the enamel even though the load was the same. Knoop hardness readings for enamel are usually from 170 to 200. Corresponding hardness readings for dentin vary from 35 to 55. Hardness will of course vary for individual teeth and for different areas in the same tooth.

Dental caries is dependent, to a large extent, upon the solubility of the enamel. The more insoluble the enamel, the more resistant it is to the action of acids which are liberated by the bacteria. Active decay with cavity formation is undoubtedly preceded by the slow dissolving of the enamel surface. For this reason, it is of fundamental importance to study the mechanism governing the solubility. The relationship of the solubility of enamel and the pH of the surrounding medium may be described in terms of the solubility product (*3*).

Since $Ca_3(PO_4)_2$ is apparently the main unit of the complex apatite salts which make up the composition of enamel, the solubility may be approximated in terms of this compound. According to the well-known solubility product principle, the following relationship holds true when calcium phosphate is in equilibrium with its solution:

$$[Ca^{++}]^3 \times [PO_4^{=}]^2 = K_{sp}$$

If the product of $[Ca_3^{++}]^3 \times [PO_4^{=}]^2$ is momentarily increased over the value of K_{sp}, precipitation of $Ca_3(PO_4)_2$ will result; but if the product is reduced below K_{sp}, solution of the $Ca_3(PO_4)_2$ will proceed until the product is again increased to K_{sp}. It thus follows that the solubility of enamel is to a large degree dependent upon the concentration of Ca^{++} and $PO_4^{=}$ in the surrounding medium. In this connection, the work of Karshan and Rosebury (*4*) is interesting. They determined the concentration of calcium and phosphate which was necessary to prevent the solubility of enamel at various pH levels in buffered solutions of lactic and succinic acids. For example, at a pH of 7.0, less than 1 mg. of phosphate per 100 cc. was required to inhibit the solution of the enamel, yet 200 mg. were required at a pH of 4.0. Thus, as the pH is lowered, additional calcium or phosphate is needed in the surrounding

medium in order to inhibit solubility. When sufficient concentrations of the two are present in the surrounding medium of the tooth, the pH can be quite low without danger of dissolving the enamel.

This rate of solution of the enamel will be governed not only by the pH, the Ca^{++} and $PO_4^{=}$ concentrations, but also by the type of acid present (*5*). We have found that tooth crowns which were stored in solutions of orange juice, lemon juice, coca-cola, and many other similar products lose as high as 20 per cent of their original weight after 20 days immersion. Fortunately, such materials do not remain in contact with the teeth for that long a period of time, yet it does show the effect that low pH can have upon the enamel.

The content of Ca^{++} and $PO_4^{=}$ in the saliva is probably always great enough to prevent solution of the enamel within the range of pH that does occur in saliva. It is a highly buffered medium, with a pH ranging from 6.8 to 7.2 (*6*), and thus aids greatly in neutralizing acids taken into the mouth and those formed in the oral cavity. This may explain why tooth decay usually takes place in the more protected parts of the enamel which are not so thoroughly bathed with saliva.

FLUORINE

In the past many different methods have been presented for the control of tooth decay, including use of certain vitamins, bone meal, diet, etc. The most recent theory, and apparently the most promising, is the incorporation in the developing enamel of ingested fluorides present in the drinking water or the topical application of fluorine to the formed tooth surface. Epidemiologic and clinical studies have brought out the fact that fluorides inhibit tooth decay up to about 40 per cent of the average of samples studied. The definite mechanism by which fluorine does inhibit decay is not too well understood. Apparently the fluorine does actually substitute into the space lattice of the hydroxy-apatite, replacing the OH group and thus forming calcium fluoro-apatite. This replacement produces a more insoluble enamel.

Recent research has shown that the heavy metal fluorides, such as lead and uranium, reduced the solubility of enamel to a much greater extent than the alkali metal fluorides which were previously used. In these cases, some of the Ca in the apatite is believed to be replaced by the heavy metal, forming, for example, a lead fluoro-apatite, $Pb_3(PO_4)_2 \cdot Ca(F)_2$. Further X-ray diffraction and spectrograph studies are needed to substantiate this.

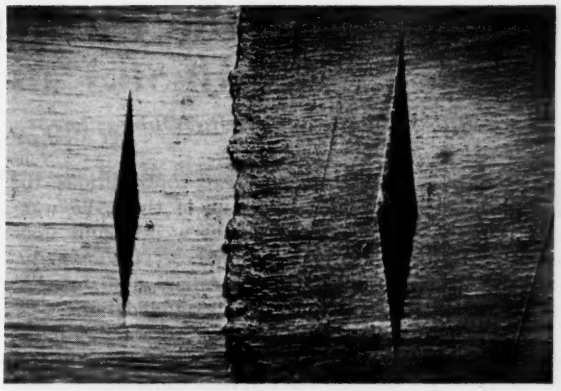

FIGURE 2.—KNOOP HARDNESS INDENTATIONS MADE ON A POLISHED SURFACE OF ENAMEL AND DENTIN AT THE DENTO-ENAMEL JUNCTION. THE SMALLER INDENTATION IN THE ENAMEL AT THE LEFT INDICATES GREATER HARDNESS.

Using the Knoop indenter for hardness, we have found further evidence of the inhibiting action of fluorine compounds. Table 1 shows the loss in enamel hardness following treatment (*a*) with a buffered acetic acid of pH 4, (*b*) with a buffered acetic acid of pH 4 plus the fluorine compound indicated. It is quite evident from these data that the enamel is protected by this replacement reaction since the loss in hardness from the original is much less in the fluorinated acetic acid.

TABLE 1

LOSS IN HARDNESS OF ENAMEL WHEN IMMERSED IN FLUORINATED AND UNFLUORINATED ACETIC ACID SOLUTION

Five groups of 12 teeth each were polished, hardness determined; each group was placed first in acetic acid, and then after determining hardness again, in acetic acid containing one of five different fluorides, with results as follow.

Buffered acetic acid at pH 4.0. Per cent change in hardness	*Buffered acetic acid plus fluoride at pH 4.0. Per cent change in hardness*	*Fluoride added*
−23.3	−8.1	1. NaF
−37.2	−2.5	2. PbF_2
−15.0	−3.7	3. CuF_2
−27.6	+6.8	4. ZnF_2
−43.8	+3.1	5. SnF_2

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MERRY CHRISTMAS

Catalysis and the Elementary Chemistry Course

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BERZELIUS made many great contributions to chemistry, but in making one of them, he was inadvertently guilty of performing a profound disservice to future students of chemistry. This anomaly arose only because he, like most scientists, first stated a generalization and then put forth the qualifications. As has happened in many another case, abstractors have lifted the generalization and left the qualifications, to the great confounding of the aspiring student.

Concerning catalysis, Berzelius, in 1835, concluded:

> It is then proved that several simple and compound bodies, soluble and insoluble, have the property of exercising on other bodies an action very different from chemical affinity. By means of this action they produce, in these bodies, decompositions of their elements and different recombinations of these same elements to which they themselves remain indifferent.

It is here that most quotations stop, but Berzelius went on to say:

> This new force, which was hitherto unknown, is common to organic and inorganic nature. I do not believe that it is a force quite independent of the electrochemical affinities of matter; I believe, on the contrary, that it is only a new manifestation of the same; but, since we cannot see their connection and mutual dependence, it will be more convenient to designate the force by a separate name. I will therefore call this force the catalytic force, and I will call catalysis the decomposition of bodies by this force in the same way that one calls by the name analysis the decomposition of bodies by chemical affinity.

In many present-day chemistry texts (12 were examined) catalysts are defined as "substances which alter the rate of a chemical reaction but are not themselves permanently changed." In most cases little attempt is made toward any type of explanation. Such a definition is not scientifically correct since catalysts do undergo "permanent change," but in two of the texts—and this is truly amazing—the definite impression is created that the catalyst does not even enter into the reaction. To these authors it is apparently less imaginative to say that the catalyst does not enter into the reaction whose rate it changes than to say that the effect is not well understood. The fact that it is inconceivable that two substances could affect one another without reacting (a contradiction in terms) apparently escapes notice. It is small wonder that a large number of students of chemistry are firmly convinced that a catalyst "speeds up a reaction but does not enter into it."

The performance of a simple experiment in the laboratory quickly convinces students that, in one case at least, the catalyst has an important role as a chemical, and not just as a bystander. For such an experiment one can turn to the classical introductory experiment on catalysis, the effect of catalysts on the decomposition of potassium chlorate.

Experiment. Heat some potassium chlorate in a test tube until oxygen just begins to evolve from the molten salt. Remove the tube from the flame and add a *pinch* of manganese dioxide. Repeat with another tube adding a *small pinch* of ferric oxide. Try calcium oxide, silicon dioxide, and cupric oxide. Compile from the text or a handbook a list of all the known oxides of manganese, iron, calcium, silicon, and copper and, on the basis of your results, try to interpret the reason for some of these oxides being catalytic for the release of oxygen from potassium chlorate. Try other oxides from the side shelf after you predict their effectiveness. (Do not use PbO_2, BaO_2, Na_2O_2, or HgO since these all release oxygen as readily as does potassium chlorate.) Is your rule sufficient or only necessary? Try Cu_2O.

In Table 1 are listed the results of the addition of various compounds to potassium chlorate which, although molten, is liberating oxygen very slowly. It may be noticed that every oxide which increases the rate of oxygen formation is of an element which can exist in various stable oxidation states, although some of the oxides which do not act as catalysts, antimony for instance, also fall into this class.

The trend in Group 5 elements in the periodic table, where P_4O_{10} and As_4O_{10} are catalysts while Sb_4O_6 and Bi_2O_3 are not, is in accord with the relative instability of the higher oxidation states of antimony and bismuth compared to those of phosphorus and arsenic. Other oxides than those listed also follow this oxidation-reduction criterion, but they are not generally available in elementary work.

It should be mentioned that the lower oxides of the positive catalysts listed are not true catalysts. Their addition to molten potassium chlorate does hasten oxygen evolution, however, for the potassium chlorate oxidizes them to the higher oxidation state. Thus, addition of red Cu_2O leads to the formation of black CuO, which is then catalytic.

It is therefore driven home to the student that in the catalysis of the decomposition of potassium chlorate by oxides a probable mechanism is the alternate oxidation and reduction of the catalyst. As a clinching experiment it might be shown that salts of the active oxides also act as catalysts, potassium dichromate for example. This indicates that it is not the crystal structure of the oxides which is the determining factor.

A catalyst is, then, a substance which, by entering into a reaction, affords a new mechanism for the reaction but causes no change in the equation for the net reaction or in the equilibrium concentrations. There is no net change in the chemical composition of the catalyst.

TABLE 1

$KClO_3 = KCl + 3/2\ O_2$

Positive catalysts	*No apparent catalysis*
CrO_3	MgO
MoO_3	CaO
MnO_2	BaO
Fe_2O_3	ZnO
Ni_2O_3	SiO_2
CuO	Sb_4O_6
P_4O_{10}	Bi_2O_3
As_4O_{10}	

Selecting a Graduate School for Training in Chemistry[1]

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HUNDREDS of G. I. Joes and Janes are asking the question, "Where shall I go for graduate work in chemistry?" When I faced the same question at the close of World War I, one of my professors offered the following suggestions: "University *A* has the most beautiful campus in America. University *B* has a wonderful school spirit. University *C* offers nothing but hard work." After a quarter of a century have we chemical educators anything more helpful to offer the veterans of World War II?

The American Chemical Society has prepared, after a decade of thorough study and investigation on the part of its Committee on Professional Training, a list of approved institutions for undergraduate training in chemistry.[2] To many of us engaged in teaching undergraduates in liberal arts colleges, the minimum requirements of the ACS committee are decidedly too professional for a student at the undergraduate level. We question the wisdom of a student's taking a minimum of 45 to 50 hours, out of his 120 hours for the Bachelor's Degree, in *any one* subject. The medical profession is not unanimous in advocating strong specialization in the natural sciences in premedical training. The chemical profession should afford the undergraduate more time for the study of the social sciences and the humanities if members of our profession are to make the contributions expected of them in our complex and confused postwar world. Has not the testimony of scientists before Congress on the pending bills on science legislation revealed to some of us our glaring lack of such broad training? And on the other hand, where will one find more eloquent appeals for scientists to move out into new positions of leadership in the world of tomorrow, than those made at the Westinghouse Forum on "Science and Life in the World?"[3]

Already educators in our institutions of higher learning are making plans to correct the wave of professionalism found in the teaching of undergraduate courses in science. There is a definite threat today to take the teaching of all first-year science courses away from the science departments of all our colleges and universities.[4] The work of the ACS Committee on Professional Training has helped precipitate this crisis.

Since we cannot expect a list of "approved" graduate departments of chemistry for some time, if at all,[1] what objective tests or standards can we suggest that a student can himself check before deciding on a graduate school? With Uncle Sam footing the bill for the veteran, the usual pressing question of finances is relegated to a position of secondary importance, where it belongs. For students, however, veterans or nonveterans, for whom the financing of graduate training is a hurdle, Dr. Alexander Silverman's study, presented at the Atlantic City meeting of the Society, on scholarships, assistantships, and fellowships granted by graduate schools, should prove helpful.

Assuming a student desires the thorough training which will enable him to pursue a research career, even though he may later find himself teaching the science or employed in a plant, a sales office, or as an executive in a chemical industry, he will do well to note where the leaders of research in America took their graduate training. The 250 starred chemists and chemical engineers in "American Men of Science," 7th Edition, is as representative a group of such leaders as one can find. The universities granting the largest number of earned advanced degrees to this group are listed in Table 1.

TABLE 1

UNIVERSITIES GRANTING ADVANCED DEGREES TO STARRED CHEMISTS AND CHEMICAL ENGINEERS IN "AMERICAN MEN OF SCIENCE," 7TH EDITION

University	*No. of masters' degrees*	*No. of doctors' degrees*
Brown	2	4
California	5	21
California Institute of Technology	1	5
Chicago	4	18
Columbia	7	9
Cornell	0	6
Harvard	18	28
Hopkins	1	15
Illinois	9	10
M.I.T.	3	8
Michigan	6	5
Ohio State	8	3
Pennsylvania	0	8
Princeton	6	9
Wisconsin	4	6
Yale	3	12
Foreign	9	51

It is significant that 16 American universities were selected as the place for their training for the doctorate by two-thirds of the starred scientists. Attention is also called to the fact that 51 of the group received their doctorates in foreign institutions; this is partly accounted for by the fact that 30 of the group also took their undergraduate work in European countries.

The advantages accruing from the selection of an institution which has the reputation of attracting superior students cannot be overemphasized. The competition during student days will certainly prove more stimulating, and more than one student in such an institution

(*Continued on page 588*)

[1] Presented before the Division of Chemical Education at the 110th meeting of the American Chemical Society in Chicago, September 9–13, 1946.

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A Presentation of the Thermodynamic Functions

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THE large number of recent papers (*1–9*) on the teaching of introductory thermodynamics indicates a widespread realization of the problems involved. It is a difficult subject to teach to beginners, probably because its real difficulties come at the very beginning. The rather elusive basic concepts have to be introduced early in the course and must be adequately explained, as well as rigorously defined, if the student is to grasp the substance behind the symbols. The beginner, moreover, cannot visualize new developments in terms of the subject as a whole, and so is unable to derive assistance from its interlocking ideas and its inherent symmetries. It would seem desirable, therefore, that a general outline of the broad essentials be sketched out as soon as possible, and that it be used throughout the course as a guide in orienting and fixing new ideas. Such a first picture is necessarily incomplete and distorted, but the details can be more easily filled in after it has been shown in a general way how the main parts fit together.

The present paper outlines a method for introducing the fundamentals of the subject to a class in undergraduate physical chemistry. It has been used by the writer for the past ten years and seems to have certain advantages over a more strictly mathematical approach. It is based on a diagrammatic representation of functions and variables and, in some respects, resembles a scheme briefly outlined by Becher (*1*). The central idea is to keep as much as possible of the subject in view at all times so that concepts already grasped will help with the understanding of new developments.

Thermodynamics is concerned chiefly with the transfer of energy from one system to another and with the transformation of energy from one form to another. It deals only with systems at equilibrium and is concisely summed up in two laws, the first of which is concerned primarily with energy transfer and the second with energy transformation. Heat and work are the only forms in which energy passes from system to system, and the conversion of heat into work is the only energy transformation that is at all restricted.

The first law states that the internal energy, U, of a system can change only to the extent that it exchanges heat, q, and work, w, with its surroundings and may be expressed as

$$U_2 - U_1 = \Delta U = q - w$$

The internal energy is a property of the system so that the ΔU of the process is uniquely determined by the initial and final states of the system. These states, however, have no part in determining q and w which represent energy in transit between systems and have no meaning when applied to an equilibrium state. The individual values of q and w are therefore no concern of thermodynamics, except in the limiting case where the process is a sequence of an infinite number of equilibrium states, *i. e.*, when it is reversible. In such a process q and w have unique and maximum values determined by the initial and final states of the system itself and thus are quantities that can be handled by thermodynamics.

If a system undergoes an internal energy change while at the same temperature as its surroundings, energy is transferred solely as work since a temperature difference is necessary for transfer of heat. That part of the internal energy that can be removed from the system at constant temperature may thus be regarded as "free" energy, *i. e.*, energy free to become work. The energy that is now left in the system can be removed only as heat and, in a sense, may be regarded as energy more closely related to heat than to work. On its appearance as heat outside the system it may, of course, be converted into work to the extent permitted by the second law; the conversion would be complete if a continuum of reversible engines all the way to absolute zero were available. Thus, while it is not permissible to describe a system as possessing so much work and so much heat, it is convenient to distinguish between that part of the internal energy that may at any temperature be removed as work and that part which must be removed as heat. These energies are single-valued functions of the variables which define the state of the system.

As might be expected, the most important variables of thermodynamics are the intensity and capacity factors of energy. These occur in pairs of such dimensions that their product is energy, *i. e.*, work or heat. They are properties usually more accessible to direct measurement and control than energy itself and are therefore the only variables to be taken experimentally as independent. Any kind (mechanical, electrical, or chemical) of work (force $\times$ displacement) may in principle be completely converted into any other kind, and so may be expressed in terms of any desired pair of intensity and capacity factors. In thermodynamics pressure and volume are often the most convenient pair to use. Heat as such is represented as the product of a heat capacity and a temperature change.

As already pointed out, the heat and work quantities of most significance to thermodynamics are those characteristic of reversible changes of internal energy. The "free" portion of this energy is directly convertible at constant temperature into work and may be expressed as such. The isothermally unavailable remainder, however, is neither heat nor work while it is still in the sys-

tem, and a special capacity factor, the entropy, S, must be introduced for it. For present purposes entropy is taken to be a quantity as fundamental as temperature, the intensity factor, and is regarded as sufficiently defined by the equation

$$S = Q/T$$

where Q is the heat transferred reversibly, *i. e.*, across an infinitesimally small temperature gradient, at temperature T. S is a single-valued function of the state of the system, and, for a nonreversible heat transfer, $S > q/T$. For an infinitesimal reversible process the first law thus takes the form

$$dU = TdS - PdV$$

and to represent the total energy of a system or the maximum work or heat of a process, it is necessary to use only two intensity factors, P and T, and two corresponding capacity factors, V and S.

These four variables P, V, T, and S, are so connected through the equation of state and the heat capacities that it is necessary to fix only two of them in order to define the state of a one-component system. There are thus six choices regarding the pair that may be taken as independent—PV, PT, PS, VT, VS, and TS. The choice actually made in any particular case will in general be determined by the data and objectives of the problem itself.

Since energy can be expressed as the product of an intensity factor and a capacity factor, it is possible to represent the energy of a system at equilibrium by an area on a diagram in which corresponding pairs of factors are generalized coordinates. In Figure 1 the ordinates and abscissas represent, respectively, intensity and capacity factors, and the area $a\,a'd'd$ stands for the enthalpy or total energy, H, of the system. Since the absolute value of H is indeterminate, this area is to be taken as the difference in enthalpy between two equilibrium states, of which one is an arbitrary zero. This diagram is not to be confused with the usual PV or TS diagram in which each line denotes a process and each point an equilibrium state; it merely represents the sum of the products of the conjugate intensity and capacity factors pertaining to the state of the system.

Part of the total energy of a system is internal energy, U, and part is external energy. The latter is the energy that a system may possess by virtue of its position in a potential field. In the simple systems considered here, this potential is uniform (*i. e.*, hydrostatic) pressure so that the external energy is PV. It may be visualized as being equivalent to the work necessary to expand the system reversibly from zero volume to the existing volume against a pressure infinitesimally smaller than its own. It is represented in Figure 1 by the area $cc'd'd$.

It has already been stated that the internal energy of a system in equilibrium at a given temperature may be divided into a part which is "bound" or latent and a part which is "free." This isothermally available energy can be completely converted into work so that it may be descriptively called maximum work or work content. It is represented in Figure 1 by the area A. The isothermally unavailable portion of the internal energy has already been designated as TS, and the corresponding area is so labelled in the diagram. The maximum work, A, is part of the internal energy, U, and does not include the external energy, although the latter also is to be regarded as isothermally available.

These three energies—external free energy, internal free energy, and internal bound energy—account for all the energy of the system, and it is not strictly necessary to introduce other quantities. It is often convenient, however, to lump together the two internal energies (bound and free) and the two free energies (internal and external) and to deal directly with the totals U and G. It is to be noted that G bears the same relation to A that H bears to U. The areas corresponding to these energies are indicated in the diagram.

It has already been pointed out that it is impossible to separate the energy of a system into so much heat and so much work. The diagram, therefore, is not a description of the system itself nor of its energy content; it represents what can be done with the energy when the system goes from one equilibrium state to another.

The six energy quantities represented as overlapping areas on the diagram are the enthalpy or total energy,

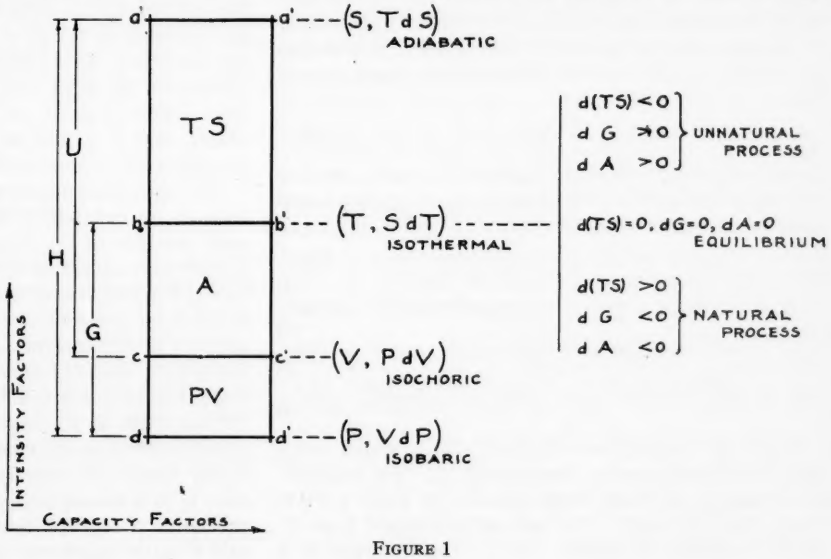

FIGURE 1

H; the internal energy, U; the Gibbs free energy, G; the Helmholtz free energy or maximum work, A; the isothermally unavailable energy, TS; and the external energy, PV. H, U, G, and A are the so-called thermodynamic functions or potentials. Each may be expressed in terms of the others (and of TS and PV) by simple inspection of the diagram. Among the relationships so obtained are

$$H = U + PV$$
$$U = A + TS$$
$$G = A + PV$$

which are often introduced abruptly as isolated equations defining the functions. The latter are thus apt to appear as unduly artificial, and the simple interconnections among them tend to be obscured.

Each of the six energies (areas) is a single-valued function of two of the four variables T, S, P, and V, any one of the six pairs of which may be taken as independent. The differential of each function may thus be equated to the algebraic sum of two terms, each containing the differential of one of the independent variables, and each term, being an energy, must be the product of conjugate intensity and capacity factors. As there are only two pairs (TS and PV) of such factors and as only two variables are independent, each differential equation must involve two terms in TS, or two terms in PV, or one term in each of TS and PV.

The diagram is explicit regarding the variables and equations for the external energy and the unavailable energy, *i. e.*,

$$d(PV) = PdV + VdP$$
$$d(TS) = TdS + SdT$$

In these cases the energy factors are also the independent variables. Equations for dU, dH, dA, and dG may be set up by noting how a change in the area corresponding to the function affects the TS and PV areas of the diagram, and by retaining only those two of the four terms so obtained that have any physical significance. For example, an increase in U means that TS increases (aa' moves up) and/or PV decreases (cc' moves down), *i. e.*,

$$dU = d(TS) - d(PV) = TdS + SdT - PdV - VdP$$

In this way equations (1) to (4), in which each term has its proper sign and in each of which there are two terms equal to zero, may be set up.

$$dU = d(TS) - d(PV) = TdS + SdT - PdV - VdP \quad (1)$$
$$dH = d(TS) + d(PV) = TdS + SdT + PdV + VdP \quad (2)$$
$$dA = -d(TS) - d(PV) = -TdS - SdT - PdV - VdP \quad (3)$$
$$dG = -d(TS) + d(PV) = -TdS - SdT + PdV + VdP \quad (4)$$

There are various ways of determining which terms are of no significance in these equations. For example, a comparison with the first law, $dU = TdS - PdV$, shows that SdT and $-VdP$ are to be dropped from (1) which becomes (5) below. From the diagram it is evident that a change in U does not involve a change in G—the two areas have no common boundary. Thus for a process to which equation (4) applies, $dU = 0$, so that $TdS = PdV$, and (4) is reduced to (8). According to the diagram also, a change in the PV part of G brings about the same change in H, *i. e.*, equations (2) and (4) retain the term VdP. In the same way it may be seen that (1) and (3) keep $-PdV$, (3) and (4) keep $-SdT$, and (1) and (2) keep TdS.

$$dU = TdS - PdV, \text{ independent variables } S \text{ and } V \quad (5)$$
$$dH = TdS - VdP, \text{ independent variables } S \text{ and } P \quad (6)$$
$$dA = -SdT - PdV, \text{ independent variables } T \text{ and } V \quad (7)$$
$$dG = -SdT + VdP, \text{ independent variables } T \text{ and } P \quad (8)$$

It will be noted that each of the four horizontal energy boundaries of the diagram corresponds to one of the four terms TdS, SdT, PdV, or VdP in equations (5) to (8) and may be identified with one of the four variables. For example, it is evident from equation (8) that an increase in G means a decrease ($-SdT$) in the TS area and an increase ($+VdP$) in the PV area, that the line bb' corresponds to the term SdT and may be regarded as representing the variable T, and that dd' stands for the term VdP and the variable P. The lines aa', bb', cc', and dd' may thus be identified, respectively, with S, T, V, and P, and, if stationary during a process, signify that it is, respectively, isentropic (adiabatic), isothermal, isochoric, or isobaric. These correlations are indicated in the middle section of Figure 1, which thus becomes a useful mnemonic for setting up various thermodynamic relationships.

These four differential equations (5) to (8) are the basis of the whole thermodynamic structure and from them may be derived the five hundred million or more formulas of the subject. Each equation is in effect a combined statement of the (conditioned) first and second laws, so that no one of them is to be taken as more fundamental than any other; each one completely defines the system, and each of the four sets of quantities may be expressed in terms of the differential coefficients of any other set. It is thus obvious that they are different aspects of the same thing (the energy of the system) from different points of view (the independent variables), and it would seem desirable that this fundamental unity be kept constantly before the student.

The equivalent nature of the different tests for equilibrium in an isolated system may be readily visualized with the help of the diagram. In considering internal equilibrium, attention centers on the partition (bb') between free and bound energy, since this is the only subdivision of internal energy that may be made. On account of the one-way nature of the heat-work transformation, the only changes that can take place spontaneously in a nonequilibrium state are those in which TS increases at the expense of G. At equilibrium all possible spontaneous changes have been completed, and to say that TS is a maximum is the same thing as saying that G is a minimum. Moreover, it follows from the preceding paragraph that the conditions that U, H, A, and G have minimum values are equally fundamental

tests for equilibrium. It is for this reason that they are called potentials. It follows also that if no natural, *i. e.*, spontaneous, change [$d(TS) > 0$; dG, etc. < 0], and no unnatural [*i. e.*, contrary to the second law] change [$d(TS) < 0$; dG, etc. > 0] is possible, the only changes that can take place at equilibrium must be reversible [$d(TS) = 0$; dG, etc. $= 0$].

For an infinitestimal change at equilibrium, therefore, equations (5) to (8) become, respectively,

$$TdS = PdV \quad (dU = 0) \tag{9}$$
$$TdS = -VdP \quad (dH = 0) \tag{10}$$
$$SdT = -PdV \quad (dA = 0) \tag{11}$$
$$SdT = VdP \quad (dG = 0) \tag{12}$$

In terms of the diagram these are the boundary shifts necessary to keep the energy areas in question from changing, *e. g.*, if $dA = 0$ and $cc'(PdV)$ moves to increase A and decrease PV, then $bb'(SdT)$ must move an equal distance to decrease A and increase TS. For equilibrium, however, all four potentials must remain unchanged at their minimum values during an infinitesimal process, *i. e.*, a shift in any boundary calls for compensating shifts in the other three, and it would plainly be convenient to have an expression for each choice of independent variables, stating explicitly how a change in any variable affects all the others.

The four so-called Maxwell relations, which are shown in Figure 2, do just this. Like equations (9) to (12) they apply only to changes at equilibrium and so have dropped the functions and retained the variables. They are so easily derived from (9) to (12) by applying the fundamental equation of partial differentiation that no attempt is made here to force them from Figure 1. They are, however, readily derived and easily visualized in terms of the diagram which can serve as a mnemonic here also.

The diagram is frequently a help in the derivation of equations and the visualizing of "effects" in that it presents the framework of the subject as a background against which details appear in their proper places and proportions. A few examples may be given:

1. For equilibrium between two phases, *e. g.*, liquid and vapor, $dG = 0$, so that

$$VdP = SdT$$

and

$$\frac{dP}{dT} = \frac{S}{V} = \frac{\Delta H}{TV} = \frac{\Delta HP}{RT^2}$$

which is the Clausius-Clapeyron equation. If a definite quantity, *e. g.*, one mol, is evaporated reversibly and isothermally, aa' moves up to increase TS and H by an amount equal to ΔH, the total latent heat of evaporation (supplied by the thermostat); bb' remains stationary; cc' moves up to increase PV and decrease A by an amount equal to the increase in external energy while dd' remains unchanged. The difference between the increase of the TS area and that of the PV area is thus the internal heat of evaporation.

2. Reversible galvanic cells are important thermodynamic systems. If no gases are involved in the cell

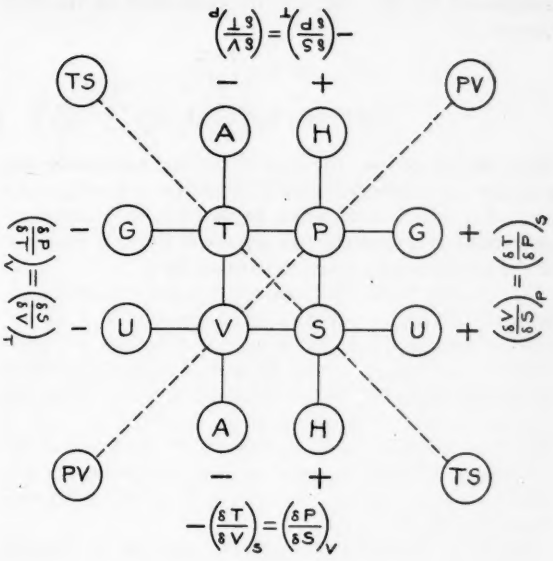

FIGURE 2

reaction, PV is negligible, cc' coincides with dd' to give, say, cd', and the equations

$$\Delta G = \Delta H - T\Delta S$$

and

$$\Delta A = \Delta U - T\Delta S$$

become identical. Referring such a thermostated cell to the diagram, it is evident that if aa' is stationary ($\Delta S = 0$), the decrease of internal energy is equal to the decrease of free energy, since the common boundary cd' moves up as the cell discharges. If aa' moves down at the same time as cd' moves up, the decrease of free energy will be less than the decrease of internal energy; some of the latter is "lost" as heat to the thermostat. If aa' moves up while cd' moves up, the decrease of free energy is greater than the decrease of internal energy, and the cell would cool if it did not absorb heat from the thermostat. The movements of aq' are thus to be identified with heat flow and entropy changes and indicate whether the reaction is endothermic or exothermic. Since entropy is, in general, more difficult to deal with than free energy, it is often desirable in the course of a problem to change from one to the other. From Figure 1, $-SdT$ is equal to dG at constant pressure, or to dA at constant volume, *i. e.*,

$$-\Delta S = \left(\frac{\delta G}{\delta T}\right)_P \text{ or } \left(\frac{\delta A}{\delta T}\right)_V$$

Substituting for $-\Delta S$ in

$$\Delta G = \Delta H - T\Delta S$$

and

$$\Delta A = \Delta U - T\Delta S$$

gives the Gibbs-Helmholtz equations in which entropy

is replaced by the temperature coefficient of the free energy.

$$\Delta G - \Delta H = T\left(\frac{\delta G}{\delta T}\right)_P$$

$$\Delta A - \Delta U = T\left(\frac{\delta A}{\delta T}\right)_V$$

Here also, of course, the sign of the last term indicates whether the free energy change is greater or less than the heat of reaction. The point to stress in chemical thermodynamics is that the free energy is the true measure of the tendency of a reaction to take place.

3. In the Joule-Thomson effect a gas expands adiabatically through a porous plug at constant H, *i. e.*, aa' and dd' remain fixed. Moreover, in this case A is pressure-volume energy, so that bb' and cc' coincide and may be designated as bc', the movements of which represent the only energy transformations involved in such a throttling process. If bc' moves up, PV increases at the expense of TS and, the process being adiabatic, the gas cools; if the gas is above its inversion point, bc' moves down and the gas heats.

While the presence of symmetry may not be of much help in the understanding of a phenomenon, it certainly makes a structure or system more interesting, satisfying, and easy to remember. The overlapping of the energy areas of the diagram in Figure 1 tends to obscure the striking symmetry that prevails among the functions and variables of thermodynamics. In a diagram of the type shown in Figure 2, however, this symmetry is clearly brought out, and the essential completeness of the system is emphasized (see also *3*). In this scheme the variables are shown connected in pairs to their corresponding functions. The fundamental differential equations (5) to (8) may be taken from this figure by noting that the positive and negative signs adjacent to the functions refer to the term containing the nearest independent variable, *e. g.*,

$$dU = +TdS - PdV$$

The scope of the underlying symmetry receives further emphasis from the fact that, by rotating the figure through 90° angles, each of the four Maxwell relationships may be picked out in a manner which is obvious from the diagram.

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SELECTING A GRADUATE SCHOOL FOR TRAINING IN CHEMISTRY (*Continued from page 583*)

has reported that he learned as much from the grilling of fellow students in a pre-Ph.D. club as from the faculty. Furthermore, the larger achievements of one's superior classmates will continue to exert its driving force for greater personal effort throughout life. And, finally, one often gains as much through thoughtful acts of friends in high places as through his own conscious efforts.

The caliber of the faculty must ever remain a determining factor in the selection of a graduate department. The prospective student can check for himself how many of the research staff are starred men in "American Men of Science," or the volume and quality of their research published in the Society's journals and monographs, or what offices they have held and what honors they have won from scientific societies, etc. While every graduate department seeks to be strong in each of the major fields of chemistry, the fact remains that a department is usually stronger in one field than another. And the selection of a major professor is even more important, for he not only directs the student's research and assists him to find a job, but he remains a personal friend and scientific godfather in after years. This latter consideration has led many a graduate student to cast his lot with a promising younger member of a staff rather than with an older member with more publications and more honors.

An appraisal of the strength of related science departments is the last suggestion we offer to a student seeking entrance into the graduate school of a university. If the individual is interested in physical chemistry, he will do well to check on the physics and mathematics departments. On the other hand, if he wishes training in the biological or medical phases of chemistry, he should know something of the reputation of these departments where he will take his minor. It is not uncommon for a "minor" to become the field of major specialization in postdoctorate research; two Ph.D.'s in chemistry of my own graduate school days are now professors of physics in the same institution.

If these suggestions seem more inadequate and misleading than those given to this veteran of World War I, then there is hope that there will be available a more objective and professional guide to place in the hands of the remnant of veterans from World War III who may seek advice on what graduate school to enter for training in chemistry. It is high time that a subject so fundamental to the development of American chemistry be divorced from personal opinions, blind loyalties, and bitter prejudices.

Catalyzing the— Approach to Equilibrium[1]

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THERE seems to be no topic in freshman chemistry that presents more difficulties to students than chemical equilibrium. After trying for over 30 years to give clear answers to their questions, I have come to have a great deal of sympathy with them, realizing that the subject is inherently a difficult one. There is evidence that it sometimes bothers instructors and even writers of textbooks. Professional courtesy prevents giving examples. One may, of course, announce rules and formulas and drill students in applying them, but the teacher who does that is evading his obligation to assist his students to gain true understanding.

In an effort to treat some of the questions of practical pedagogy involved, let us discuss several examples of typical student difficulties. We inform the student that the proper way to write the solubility product for $Ca(OH)_2$ is $K_3 = (Ca^{++})(OH^-)^2$ but why, he asks, is it $(OH^-)^2$? The teacher may reply that since the equation for solution is $Ca(OH)_2 = Ca^{++} + 2OH^-$, the product of the three ions on the right must be used, $(Ca^{++})(OH^-)(OH^-)$. If the student accepts this explanation, he may then ask the reasonable question, as many of mine have, whether in a saturated solution of $Ca(OH)_2$ in pure water, where $(Ca^{++}) = 0.02$, and $(OH^-) = 0.04$, one should write accordingly $K_3 = 0.02 \times 0.02 \times 0.02$? Any instructor can answer dogmatically, "No, one writes $0.02 \times \overline{0.04}^2$." But can he make the reason for the latter choice clear?

Or, again, if the student should ask, as he may, "Why is the equilibrium pressure of CO_2 over a mixture of $CaCO_3$ and CaO independent of the relative amounts of the two solid phases?" what is the instructor to answer? Or, to consider a third case, how many instructors can themselves give a satisfactory explanation of the fact that although carbonic acid is weaker than acetic, a molar solution of sodium bicarbonate is less alkaline than molar sodium acetate?

The basis of such difficulties lies, I believe, in the statistical basis of equilibrium; the student has to cultivate an imagination able to visualize the simultaneous, different behaviors of molecules of several different species. This is much harder, both to see and to illustrate, than the reactions of individual molecules. It would be easier if simple probability theory had a larger place in elementary mathematics. This could lend interest to that subject since so many human problems involve degrees of probability rather than true or false logical propositions.

The instructor should, of course, be able to state clearly the actual processes going on in a dynamic equilibrium, such as that between acetic acid and its ions in aqueous solution, but teachers should never forget that everybody finds it difficult to solve problems in unfamiliar terms; it is in practice easier to compute the price of five oranges at 36 cents per dozen than the price of five squirnks if 12 of them cost 36 shekels. Few of us would be quite so helpless as the poor woman on the radio quiz program who excused herself from naming a "city in Europe directly south of the North Pole" on the grounds that she had "never been to Europe," but we are all a little like that; most chemists would tackle a problem in which t stands for temperature with far more confidence than if it stands for time, so let us help out our struggling freshmen wherever possible, even though our best illustrations must admittedly be imperfect.

A dance offers a partial analogy with a chemical equilibrium, such as the ionization of a weak acid. If $HC_2H_3O_2 \rightleftharpoons H^+ + C_2H_3O_2^-$ is a little mysterious[2] and unreal during the first term of the freshman year, gent. + lady $\rightleftharpoons$ gent.-lady (dancing couple) is familiar enough to most. The reaction takes place in "both directions" more or less simultaneously. There is "disordered motion," both linear and rotational, and energy may even be expended in vibration. There are, at any instant, a certain number of "wall flowers," or lone "ions," but not always the same individuals, and their number will vary with the number of opposite partners present; if more "gents" arrive, the number of unpaired ladies diminishes, but the type of the reaction is not altered, only the number of couples. One never sees the reaction: 2 gents. + lady $\rightleftharpoons$ $(gent.)_2$-lady, no matter how many of the former are present. This is worth carefully noting since students, if asked to represent what happens when sodium acetate is added to the acetic acid equilibrium, often add $C_2H_3O_2^-$ not to the solution but to one side of the equation, thereby spoiling it as an equation.

The effect of diluting acetic acid can be illustrated by throwing open an adjacent ballroom, permitting a crowded dance to spread over more space. In the interchange between dances partners will then be harder to locate, and the percentage of "ions" will be, temporarily, greater. Ingenious instructors can think of

[1] Presented before the Division of Chemical Education at the 110th meeting of the American Chemical Society in Chicago, September 9–13, 1946.

[2] An instructor who insists that hydrogen ion is definitely H_3O^+ must use a different and a simpler process to fit this illustration.

other ways of displacing the equilibrium, *e. g.*, by the escape of a number of individuals of one of the species to consult the mirror in the dressing room.

The illustration can be only qualitative, but it is very important, in my opinion, for the student to get a clear qualitative concept of equilibrium before resorting to quantitative formulations. Not all will agree with this; orthodox instruction in physics nearly always consists in deriving formulas and turning them over to the student to apply, often in a rather mechanical way. I regard this as, first of all, poor pedagogy, for our object should not be limited to getting the right answers; second, impractical, since we want qualitative answers far more often than quantitative; and, third, misleading, since the theory is often qualitatively correct but quantitatively very inaccurate, as always where concentrations of ions are used in place of their more accurate activities.

But to return to the solubility product of $Ca(OH)_2$. The solid in equilibrium with its saturated solution is always dissolving at one point and being reformed at another, as shown by the growth of fine crystals. The redeposition involves the union of one Ca^{++} and two OH^-. If the concentration of OH^- is doubled, the rate of deposition is quadrupled until the concentration has become one-fourth as large, thus restoring the rate to equal again the rate of solution. But ions are difficult to imagine, so let us consider hunters out to shoot deer in a large area of some National Forest. Each hunter is allowed to shoot two deer. How will the rate at which hunters get their "limits" depend on the "concentration" of the deer in the region? It is obviously proportional to the "concentration" of hunters. If n is the average number of deer per square mile, the speed with which a hunter locates and shoots his first deer is proportional to n. There are then $n - 1$ deer left, so that the speed with which he gets his second deer is proportional to $n - 1$. He gets his pair at a rate proportional to $n(n - 1)$. If n is large, this is practically n^2. It is not $(n/2)(n/2)$; the chance of getting one deer is dependent on the total number of deer still alive, since any one of them will serve the purpose, but shooting one does little to decrease the chance of getting within range of the second. It is not necessary, we see, to have a "triple collision," which is very improbable, in order for the law to hold, for $A + B \rightarrow AB$ followed by $AB + B \rightarrow AB_2$ gives also the statistical probability corresponding to $n_A n_B^2$.

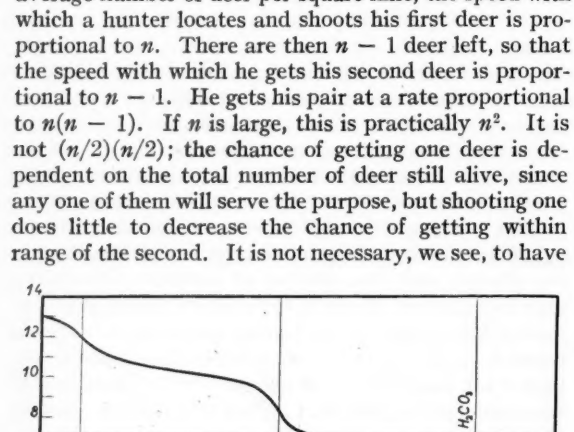

FIGURE 1. CHANGE IN pH ON ADDING TO 1 LITER OF 1 *M* $NaHCO_3$, FRACTIONS OF A LITER OF (*a*) 1 *M* NaOH (TO THE LEFT OF 0), AND (*b*) 1 *M* HCL (TO THE RIGHT)

Let us consider, finally, the problem of the alkalinity of a solution of sodium bicarbonate; why is it less than one of sodium acetate? There are two ways of representing the true situation. If HCO_3^- behaved only in its one role as the ion of a weak acid, uniting with some of the H^+ of water, thus,

$$HCO_3^- + H_2O = H_2CO_3 + OH^-$$

the OH^- would be liberated to a higher concentration than in the case of acetate ion, as we can infer from the fact that plain soda water is far less sour than vinegar; but HCO_3^- has a second role. It is not only the ion of the weak acid, H_2CO_3, but it is itself an acid, dissociating thus:

$$HCO_3^- = CO_3^{--} + H^+$$

But since $H^+ + OH^- = H_2O$, these two processes assist each other, giving as a net result

$$2HCO_3^- = H_2CO_3 + CO_3^{--}$$

with H_2CO_3 and CO_3^{--} nearly, but not quite, equal, since an indicator test shows the concentration of OH^- in 1 *M* $NaHCO_3$ to be 2×10^{-6} instead of 10^{-7}.

Now let us put the case a little differently. Among a lot of bicarbonate ions, as here represented, one may lose its H^+ which may later attach itself to one of the HCO_3^-, since there are so many of them, rather than rejoin the CO_3^{--}, in spite of the greater attraction of the latter when on equal terms.

$$\begin{array}{l} HCO_3^- \\ HCO_3^- \\ HCO_3^- \\ CO_3^{--} \\ H^+ \swarrow\ HCO_3^- \\ \quad\ \searrow HCO_3^- \end{array}$$

It is, of course, desirable to have an idea of the concentrations present in such a solution as well as in solutions of CO_2 and Na_2CO_3. The following summary is based upon experimental data for 25° readily verifiable.

Solution	*Present at equilibria*
CO_2 at 1 atm.	$H_2O + CO_2 = H_2CO_3 = H^+ + HCO_3^-$ $0.034\ M \quad 10^{-4} \quad 10^{-4}$ (since CO_3^{--} is very small)
1 *M* $NaHCO_3$	$2HCO_3^- = H_2CO_3 + CO_3^{--}$ $0.98\ M \quad 10^{-2} \quad 10^{-2}$
0.5 *M* Na_2CO_3	$CO_3^{--} + H_2O = HCO_3^- + OH^-$ $0.49\ M \quad 10^{-2} \quad 10^{-2}$

Nature has been considerate in making four of these concentrations all equal to 10^{-2}. We can now ask a number of interesting questions involving shifts in these equilibria, such as the effect of adding Ca^{++}, boiling, diluting, etc.

After, but not before, developing skill in handling and understanding such qualitative questions, it is time for students who are not appalled by a little algebra to learn something about quantitative treatment. The first ionization constant of carbonic acid can be calcu-

ated from the first set of experimental figures above.

$$K_1 = \frac{(H^+)(HCO_3^-)}{(H_2CO_3)} = \frac{10^{-4} \times 10^{-4}}{0.034} = 3 \times 10^{-7}$$

More accurate data raise this to 4.3×10^{-7}, but the difference has little practical significance when temperature is not narrowly controlled.

The ionization constant for HCO_3^-, K_2, can be calculated from the above figures for 0.5 *M* Na_2CO_3. Its value is to be found by inserting the proper figures for the concentrations contained in the expression:

$$K_2 = \frac{(H^+)(CO_3^{--})}{(HCO_3^-)}$$

These values are as follows:

$$(H^+) = 10^{-14}/(OH^-) = 10^{-12}$$

$(CO_3^{--}) = 0.5$, since only one per cent of it is changed to HCO_3^-.

$(HCO_3^-) = (OH^-) = 10^{-2}$, since the hydrolysis equation shows that one OH^- is liberated for every HCO_3^- formed. Therefore,

$$K_2 = \frac{10^{-12} \times 0.5}{10^{-2}} = 5 \times 10^{-11}$$

Note that it is not necesssary to introduce the constant for the hydrolysis equation as some teachers feel they must do.

Having K_1 and K_2, it is now easy to show that they yield the figures given for the equilibrium

$$2HCO_3^- = CO_3^{--} + H_2CO_3$$

for

$$\frac{K_2}{K_1} = \frac{(H^+)(CO_3^{--})}{(HCO_3^-)} \times \frac{(H_2CO_3)}{(H^+)(HCO_3^-)} = \frac{(CO_3^{--})(H_2CO_3)}{(HCO_3^-)^2} = \frac{3 \times 10^{-7}}{5 \times 10^{-11}} = 0.6 \times 10^{-4}$$

Since (H_2CO_3) and (CO_3^{--}) are nearly equal, each is 0.8×10^{-2} when $(HCO_3^-) = 1$.

There are always a few intelligent students in every class whose appetite for chemistry is almost unlimited, and it is important to offer them plenty of solid food. Here are a few suggestions.

1. Reproduce the curve shown in Figure 1 by plotting a series of values of pH calculated by the aid of K_1 and K_2 for various ratios of (H_2CO_3) to (HCO_3^-) and (HCO_3^-) to (CO_3^{--}) for the respective branches. To get the value for pure 1 *M* HCO_3^- shown in the curve, it is necessary to note that (CO_3^{--}) is not exactly equal to (H_2CO_3), as assumed above, but rather that

$$(H^+) + (CO_3^{--}) = (OH^-) + (H_2CO_3)$$

2. Calculate the ratio $(H_2CO_3)/(HCO_3^-)$ in blood from its pH = 7.4.

3. The solubility product of $CaCO_3$ is 5×10^{-9}. The partial pressure of CO_2 in air is 3×10^{-4} atmospheres. From these figures and data selected from the above, calculate (*a*) the number of grams of $CaCO_3$ that could be dissolved by one cubic meter of natural surface water flowing over limestone rock, and (*b*) the number of grams of soap, considered as $C_{18}H_{32}O_2Na$, that this hard water could destroy.

4. The value of (Ca^{++}) in sea water is approximately 10^{-2} *M*. What pH must an oyster be able to establish in order to makes its shell from this Ca^{++} and the CO_2 dissolved in sea water? (*Cf.* Problem 3.) This is a hard one, but a student of chemistry should try to know as much as an oyster about pH.

5. Clinch the whole drill by applying it to H_2S, with $K_1 = 10^{-7}$, $K_2 = 10^{-15}$, and $(H_2S) = 0.1$ at 1 atmosphere and room temperature. Calculate (S^{--}) for H_2S in the presence of 0.1 *M* HCl, consult solubility products for FeS and CuS, and see why the latter is precipitated but not the former. Finally, explain why HgS dissolves to form HgS_2^{--} in a solution of sodium sulfide but not in one of "ammonium sulfide."

By this time, the student will know a lot about chemical equilibrium.

Now, finally, I should like to inject something controversial. Some may object to my presentation in that I have not used what many regard as the "modern" and only correct system of acids and bases. I should have written not H^+, an abbreviation for H^+ (aq.), but H_3O^+, and I should not have mentioned the taboo word, "hydrolysis." My defense is that any system of acids and bases represents not truth but convenience, appropriateness to the particular phenomena under consideration. These may involve solutions in water, in liquid ammonia, or in the favorite solvents of the organic chemist, or they may involve molten lavas. One should choose the system most appropriate to the case, just as the musician chooses the clef corresponding to the range of the particular instrument.

My late distinguished colleague, Gilbert Lewis, stated clearly, as early as 1923, the different possible acid-base systems, including the one now usually called the Brönsted system. The advantages of this system for the ordinary purposes of the organic chemist are so great as to have caused a wave of enthusiasm and to have attracted adherents who insist that it is the one modern, correct system, and must be taught as such even to freshmen. Evidently not all have appreciated the experiments of Lewis in which typical acid-base reactions and indicator changes take place with acids and solvents which can donate no protons to bases. Surely all should conclude from this that the Brönsted is not the one and only admissible system. I, myself, fully appreciate, I trust, its usefulness for the purposes for which it is appropriate, but I do not use it in teaching freshmen for two reasons: first, I regard it as better pedagogy to begin with the ordinary water system, expanding to the proton-donor-acceptor system later for the special purposes of organic chemistry; and second, because the other systems are equally "right" and so much more useful for certain purposes that the Brönsted viewpoint should not be allowed to usurp the entire field.

A teacher in another institution has stated his experience regarding the pedagogic difficulties of this approach as follows:

At our institution I have been required to present the Brönsted definitions as *the* definitions of acids and bases. To avoid confusion and doubts, the students are not supposed to be made aware of the existence of other points of view. The students have great difficulty with the Brönsted concept. We make the presentation as simple as possible, then conclude that the students haven't grasped it, so we go over it again and again. We spend an inordinate amount of time on it—and then recognize that the students have digested very little of it, so we ask practically nothing about it in the examinations. For the sake of simplicity we practically ignore the Brönsted concepts in the remainder of the course. All in all, the result is a mess. I believe the majority of our freshman staff agrees with me in this conclusion, and I believe the Brönsted theory is ignored completely in the analytical courses that follow. I am decidedly *not* in favor of going Brönsted in the freshman courses.

The "confirmed Brönstedians," who would criticize me for writing H^+ (aq.), or H^+ for short, are not, I find, always consistent with respect to other ions. For example, Sn^{++++} in aqueous solution is as assuredly an acid as is "H_3O^+"; why should the water not be indicated? The reason is, I suspect, that they do not know exactly how much water is attached to stannic ion, and they know that they do not know. They should realize that they are equally ignorant regarding the hydration of hydrogen ion; with hydrogen bonds being formed and broken continually in water, how can anyone be sure that a proton isolates and attaches itself to only one water molecule?

I note a further illusion—namely, that the writing of an equilibrium in the form, acid = proton + conjugate base, allows us to express the true strengths of acids, apart from the solvents used. This is only roughly true, because the particular solvent used affects not only the proton but the activities of the acid and the base as well. There is no such thing as a true constant for such an equilibrium divorced from the solvent.

I hope not to be misunderstood as objecting to the value and use of the Brönsted concepts, particularly in organic chemistry. What I criticize is only, first, the notion that they alone are right and, second, the poor pedagogy of *beginning* the teaching of acid-base equilibria in these terms. In the forthcoming, fifth edition of my "Principles of Chemistry," I have endeavored to give, in a whole chapter devoted to "Systems of Acids and Bases," as full and sympathetic account of the Brönsted system as possible, and I should regret being considered as an out-and-out "anti-Brönstedian."

Creative Chemistry Award

The Cleveland Section of the American Chemical Society is sponsoring a contest for a "Creative Chemistry Award." The general plan of the contest may be of interest to other groups; complete details may be had from the secretary of the Section, Dr. F. B. Dutton, Baldwin-Wallace College, Berea, Ohio.

A $100 Government Bond will be awarded to that student of a Senior High School who is judged to have shown the greatest originality and creative ability in constructing a model or miniature of a chemical process, reaction or principle, or of chemical equipment, apparatus or machinery, or of atomic or molecular structures. Recognition will also be given the next nine models in the order of selection by the judges.

Each Senior High School in the surrounding counties is asked to announce the award early in the fall term and encourage as many interested students as possible to enter the contest and begin working on a project. Entry is not limited to those enrolled in chemistry classes but is open to every student enrolled in the school.

Each school is to hold its own contest in any manner which conforms with the rules of the contest, and send in the name and address of the winner. The winners from each school are then to bring their models to some designated laboratory or classroom in Cleveland and have the models set up to be demonstrated. A committee of judges selected by the Cleveland Section will inspect the models and listen to an explanation and demonstration, not to exceed ten minutes, by each contestant. A statement of originality will be required from each contestant, since giving or receiving other than purely mechanical aid is prohibited.

The contestants will be entertained at a luncheon followed by trips through chemical industries or other events provided by the Cleveland Section. Following the afternoon events, a complimentary dinner for the contestants will be given by officers and members of the Section, at which time the ten best selections will be announced and the winner named. The best ten among the contestants will each be given a year's subscription to the JOURNAL OF CHEMICAL EDUCATION, and the winner of the contest will be presented a $100 Government Bond.

After the contest, the best ten models will be placed on display. The ten winning contestants will be asked to demonstrate their models at a later meeting of the Cleveland Section.

Reprints of the Periodic Classification of the Elements

Cardboard copies of the periodic table on page 603 of this issue of the JOURNAL may be obtained from the Chemical Education Publishing Company, 20th and Northampton Sts., Easton, Pa., at the following prices:

Single copies, each	$0.10
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Photography as a Basic Science

W. C. OELKE

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PHOTOGRAPHY, by the nature of its subject matter, has certain advantages as a basic science course for liberal arts students not possessed by any single one of the traditional sciences. Recent experiences in the teaching of photography have led the author to re-examine typical college offerings in the elementary sciences and to present some of the more cogent arguments for photography's being accepted more widely as one of these.

The position of the natural sciences is well established in our American colleges. Most college faculties agree that the study of science is a *must* even for the liberal arts student; hence the various science requirements found in most college catalogs.

Students in college take a science for one of three reasons: 1, direct vocational interest; 2, interest in the facts and methods of science as part of a liberal preparation for life in a scientific age; 3, to meet the formal curricular requirements for graduation without specific interest in any one science. Traditionally, the selection is from botany, chemistry, physics, and zoology, although occasionally astronomy or geology are offered. The courses are well planned, textbooks are interesting and modern, laboratories are well equipped, and the instructional level as a rule is high; but by and large we, as teachers, must admit that the work is aimed at the first group of students mentioned—those interested in a particular science as a life work, and from whom come the majors in our departments. I have no real quarrel with this emphasis in our work; we must keep even our beginning courses in science of real professional caliber if we are to deserve the name of collegiate institutions. Nevertheless, are we satisfying the interests and needs of those who elect science, either freely or under duress, as part of a liberal education? I think not, and I believe that in the immediate future we will be forced to do something about it.

One approach to the solution of this problem is the so-called "cultural" course in science, largely lecture and demonstration, which attempts to give the non-science student a "diluted" version of this or that science together with many of the facts of science which underlie our industrial civilization. This may satisfy the purely informational needs of the liberal arts student, but it cannot develop the precise habits of thinking and the objective attitude of mind which are the most valuable by-products of a scientific education. These are the very products which need most to be disseminated through the rank and file of our population in this atomic age when fuzzy thinking and prejudicial attitudes may well be fatal to us all.

The other approach is the survey course in science, which gives a broader informational view to the non-science student, but which fails at exactly the same point as the cultural course to inculcate deeply the values of the scientific method. Furthermore, it leaves the student with no very well integrated body of knowledge.

It is the thesis of this paper that a rigorous course in general photography may be made to have many of the advantages of a survey course in the sciences and at the same time meet most of the objections above mentioned.

Photography embraces many of the facts and principles of the other sciences and at the same time has a natural organization of its own. These facts make it a complete unit in contrast to the rather artificial organization of a survey course. In a broad course in photography such principles of physics as mechanics, light, heat, electricity, and even sound may be studied in direct relation to the camera, shutter, lens, film sensitivity, lighting equipment, and sound recording. The mathematics involved in such a course includes simple arithmetic, proportion, elementary algebra, and trigonometry, and may in advanced work be extended to calculus and matrix algebra in connection with the presentation of modern color theory. The place of chemistry is so obvious in relation to the whole photographic process that little comment is needed. More than a brief study of the basic laws of chemistry, behavior of solutions, properties of many inorganic and organic substances, and oxidation and reduction is fundamental. The study may be logically extended to diffusion, equilibrium, reaction velocity, complex ions, and colloids. While the biological sciences are not directly related to the photographic process, the photography of plants and animals demands some knowledge of their habits and classification and leads naturally to the desire to know more about them. For many years the magazine, *American Photography*, has maintained an extensive nature section where the life histories and detailed characteristics of various plants and animals are discussed and illustrated. In a similar way nature and landscape photography may lead to a more than cursory interest in the geographical and geological background of a region.

Photography develops the eye and hand as well as the mind. Some of the residua usually claimed for laboratory training are the development of the powers of observation, accuracy in the recording of data, manual skills, scientific objectivity, and the relation of cause and effect. This we commonly lump together under the heading, "scientific method." In most beginning science courses the experiments from day to day are short, usually unrelated to one another, and aimed at

illustrating a basic principle or demonstrating the structure or mode of behavior of some matter under consideration. The student either succeeds in obtaining the desired results or he fails; there is little opportunity to repeat the experiment or to examine the reasons for success or failure, and still less opportunity to develop skill in any one process.

In the photographic laboratory the student learns early the techniques of developing film and making paper prints. It is driven home early and by hard experience that correct techniques bring succesful and pleasing results while carelessness or lack of cleanliness results in at least partial failure. In photography the reward of careful planning, intelligent judgment, accurate measurements, and precise timing is a clear negative and a brilliant print which are a pleasure to show one's friends and to treasure for many a day. This experience stimulates interest, lifts the ego, and makes tangible the results of the scientific process. This happens not once, but time after time with increased success as the basic techniques are perfected and elaborated. On the other hand, nowhere do I know a field where inattention to details or a tendency toward slipshod work shows up sooner or is more directly traceable to its source than in the dark room. Cause and effect go hand in hand. Rigor of presentation and precision of method are immediately justified.

Photography develops powers of observation. The human eye has a tendency to see things as a unit and must be trained to observe details. How often in examining a photograph does one see obtrusive details that he never realized were present in the original scene. Experiences of this kind train the photographer to observe closely and miss no detail in foreground, principal subject, or background of the area under observation.

Photography is related more closely than other sciences to the arts. In many ways it is an art as well as a science. The principles of artistic perception and composition are inherent in a course in photography. One strives to develop a feeling for line and color. The appreciation of beauty is thus developed and heightened. One must also know people in order to photograph them well. A little applied psychology is much better in photographing a fussy baby, or a fussy grown-up, than all the scientific objectivity in the world.

Photography has vocational possibilities. The 1940 census lists 37,000 professional photographers as against 60,000 chemists. I have been unable to find data for physicists or biologists, but it is my impression that these groups are considerably smaller. Thirty-seven thousand is a good-sized professional group, and the demand for photographers, stimulated enormously by the developments coming out of the war, is bound to increase rapidly. A recent pamphlet from the Rochester Institute of Technology lists 27 different fields of photography among which are commercial, illustrative, legal, news, medical, and spectrographic photography, as well as various types of photo-mechanical reproduction. Templet photography, unknown before the war, is a new industrial technique which made history in the airplane industry and is rapidly spreading to other fields. Color photography is just a lusty infant at the moment. Photography is a growing professional field and one open to both men and women on an almost equal basis. Photography is of value to the prospective teacher, psychologist, doctor, scientist, artist, writer, or dramatist. In many fields the recording and presentation of information in the form of pictures, slides, or movies is of tremendous importance. Whether the photographic work is done directly by the individual, or merely under his direction, some photographic knowledge is essential.

Photography has a value to almost any individual as a hobby or leisure time avocation. There is scarcely an adult today who at sometime or another has not taken a snapshot and wished that he knew more about what he was doing. Whether a person finishes his own or has his photo-finishing done, knowledge of how to handle and set a camera, how to pose a subject or to compose a picture is knowledge that results in better pictures and increased satisfaction.

Summing up the points in favor of photography's taking its place in the college curriculum along with the other laboratory sciences: It gives basic training in and logical use of many of the facts of chemistry, physics, and mathematics as a part of one naturally integrated field. It stimulates interest in other widely divergent fields of knowledge. It gives rigorous training and cumulative experience in many phases of the scientific method. It has vocational value in its own right, marginal value to those training for many other professions, and long time hobby value to most others. In many ways photography is ideally fitted to serve as a basic science course for nonscience majors and as an auxiliary course for students preparing in one of the sciences.

Photography should be offered more widely as a full year course, with three hours of lecture per week and three to four hours of laboratory, and accepted on a par with the other sciences toward fulfilling laboratory science requirements in colleges and universities. Experience indicates that two two-hour laboratory or dark room periods each week, interspersed with occasional field trips, are more economical of time than one longer work period. Ninety-five standard colleges and universities in 31 states now offer at least one course in photography. In some a sequence of two or three courses is available. It is time more collegiate institutions added this field of science to their curricula.

Recent Hypotheses Concerning the Mechanism of Photographic Development[1]

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PHOTOGRAPHIC development is essentially an amplification process, and the factor of amplification can be very large. It is of the order of 10^8 in some of the more sensitive photographic "emulsions." The amount of photolytic material formed by a normal exposure of the photographic "emulsion" to the action of light is so small that it cannot be detected by the usual analytical procedures, and it cannot be seen even at the magnification supplied by the electron microscope. Yet the action of the developer amplifies this minute amount of material to the very substantial image of the developed photograph.

The light-sensitive element of the photographic film, plate, or paper consists essentially of tiny silver halide crystals, or "grains," suspended in a layer of gelatin. The discrete structure of the light-sensitive material is of primary importance. The individual grain is the unit, both for the exposure process and for the development process. The action of light upon a grain during a normal exposure results in the formation in that grain of a minute amount of silver.[2] This silver is in the form of atom aggregates, or nuclei, and is usually associated with specks of silver sulfide which were present in the grain prior to the exposure. We shall not consider here how the silver atoms happen to occur in aggregates rather than as individuals scattered throughout the grain. Several reviews of the modern theory of latent-image formation have been published in recent years,[3,4,5,6] and the theory has not undergone significant change since these publications appeared. It will suffice for our purposes to note that a grain which contains a suitable nucleus is "developable," *i. e.*, it will be reduced to metallic silver by a developer acting for a "normal" time upon the photographic material. The all-important property of the developing agent is that it can reduce such a grain *in a substantially shorter time* than it can reduce a grain which does not contain a suitable nucleus. For the most part, development instigated in one grain does not spread to neighboring grains, and the action of the reducing agent can easily be interrupted at a point where all or most of the grains which contain suitable nuclei have been completely or almost completely reduced, but reduction of the other grains (with the exception of a relatively small number of "fog" grains) is scarcely detectable.

Within certain rather wide limits, the number of developable grains per unit area, and hence the amount of silver which can be obtained on development, increases with increasing exposure. If the photographic material is given a very small, uniform exposure to photographically active light, a small percentage of the grains will be made developable. If the exposure is increased, the percentage of developable grains will be increased. If the exposure varies from area to area on the photographic material, the percentage of developable grains will vary in an analogous manner. In this way, the developer can translate varying amounts of light falling upon the sensitive layer into varying amounts of silver produced per unit area and thus translate the image or pattern of the light falling upon the photographic material into the silver image of the developed photograph.

THE MECHANISM OF DEVELOPMENT

Historically, the mechanisms which have been proposed for the development process fall into two rather broad groups. The first group includes mechanisms based on the assumption that the latent-image nuclei serve merely as condensation centers for silver formed by the reduction of silver ions. It is assumed that the latent-image material plays no part in promoting the specific reaction between the developer and silver ions. Instead, a reversible reaction takes place between the developing agent and the silver ions in solution, the concentration of the latter being determined by the solubility product of the silver halide. In the absence of nuclei upon which silver can condense, reaction quickly reaches an equilibrium. Only a very small amount of silver halide has been reduced at this point, and the silver formed remains in a supersaturated solution. When nuclei are present, however, silver condenses upon them, and the equilibrium point is never reached. Instead, more and more silver halide dissolves, the silver ions are reduced, and the silver deposits out upon the nuclei until all of the silver halide is used up. This mechanism, suggested by Ostwald and Abegg, is still given in some textbooks as a plausible one, but the evidence against it as a major factor in photographic development is overwhelming.[5] We need not consider it further here.

The mechanisms of the second group are based upon

[1] Presented before the Division of Chemical Education at the 110th meeting of the American Chemical Society in Chicago, September 9–13, 1946.

[2] *Cf.*, however, M. L. HUGGINS, *J. Chem. Phys.*, **11**, 412 (1943).

[3] WEBB, J. H., *J. Applied Phys.*, **11**, 18 (1940).

[4] MEES, C. E. K., "The Theory of the Photographic Process," The Macmillan Company, New York, **1942**, chaps. IV–VII.

[5] JAMES, T. H., AND G. KORNFELD, *Chem. Revs.*, **30**, 1 (1942).

[6] BERG, W. F., "Physical Chemistry of Latent Image Formation," *Ann. Repts. Progress Chem., Chem. Soc., London*, **39**, 49 (1942).

the assumption (well established) that the latent image nuclei in some way act to accelerate the specific reduction of silver ions. We can divide these mechanisms into two groups. Those of the first group treat the latent-image nucleus as an "electrode" which readily accepts electrons from the developer. The developing agent can give up electrons to this electrode at any point on its surface. The total reaction, accordingly, consists of at least two steps. These may be symbolized

$$\underset{\text{(nucleus)}}{n\text{Ag}} + D\epsilon \rightarrow n\text{Ag}\epsilon + D \qquad (1)$$

$$\text{Ag}^+ + n\text{Ag}\epsilon \rightarrow (n+1)\text{Ag} \qquad (2)$$

where $D\epsilon$ stands for the developing agent and D for the oxidized form. Development can occur if, and only if, the transfer of electrons to silver and the subsequent transfer of electrons from silver to silver ions occur more readily than the transfer of electrons to the silver halide crystal or to silver ions.

The mechanisms of the second group are based on the assumption that the molecules or ions of the developing agent participate directly in the reduction of the silver ions in contact with the silver nucleus. Development can occur when the reaction between silver ions and developing agent at the silver-silver halide interface requires considerably less activation energy than the reaction of silver ions and developing agent at any point out of contact with the silver.

In basic ideas, the mechanisms of the two groups are not so far apart. In the first group, the implicit assumption is made that the activation energy for reactions (1) and (2) is considerably smaller than that for direct transfer of electrons to the silver halide. In the second group, the explicit assumption is made that the reaction

$$D\epsilon + \text{Ag}^+ (n\text{Ag}) \rightarrow D + (n+1)\text{Ag}$$

or

$$(D\epsilon\ldots\text{Ag}^+\ldots n\text{Ag}) \rightarrow D + (n+1)\text{Ag}$$

occurs with smaller activation energy than the direct reaction

$$D\epsilon + \text{Ag}^+ \rightarrow D + \text{Ag}$$

or

$$(D\epsilon\ldots\text{Ag}^+) \rightarrow D + \text{Ag}$$

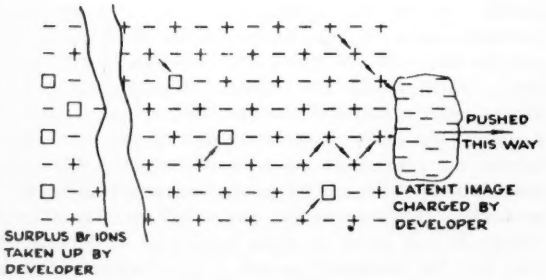

FIGURE 1. SCHEMATIC ILLUSTRATION OF BERG'S DEVELOPMENT MECHANISM, SHOWING HOW SILVER IONS FROM THE NORMAL CRYSTAL POSITIONS CAN MOVE UP TO THE NEGATIVELY CHARGED LATENT-IMAGE (SILVER) NUCLEUS

"ELECTRODE" MECHANISMS

The first mechanism belonging to this group was suggested by Gurney and Mott as an extension of their mechanism of latent-image formation.[3-6] They proposed that "the molecules of the developer hand over electrons to the metallic silver speck (latent-image nucleus) and raise its potential relative to that of the halide grain. The interstitial silver ions (*i. e.*, ions which, by virtue of thermal energy, have become displaced from their normal lattice positions into interstitial positions in the crystal) which are present in the halide are then attracted to the silver speck; they will move up to it and adhere to it. Of course, as fast as the (interstitial) silver ions in the grain are used up, new ones are formed."[7]

The accretion of silver atoms at the inner interface Ag/AgBr (inside the solid) builds up a pressure which pushes the growing silver speck outwards, thus giving rise to silver filaments. The hypothesis attempts to account in this way for the formation of silver filaments during development.[8,9]

Although the Gurney-Mott mechanism of *latent-image formation* has received strong support from experimental observations, several serious objections have been raised to the extension of this mechanism to cover the development process.[5,10] For example, no reasonable mechanism is provided for the escape of the halide ions from within the crystal. The mobility of chloride and bromide ions is at least a thousand times smaller than the mobility of silver ions.[11] For that matter, even the rate of formation of interstitial silver ions is too slow to account for the rate of development of silver bromide and silver chloride grains in the more rapidly acting commercial developers.[12] Further evidence against the Gurney-Mott scheme is found in the fact that, other things being equal, the rate of development of AgI, AgBr, and AgCl increases in that order, whereas the mobilities of the silver ions decrease in that order.[10]

Berg has avoided the difficulties of the Gurney-Mott mechanism by making the following modifications:[12]

> Imagine a latent image speck charged to capacity by electrons from the developer. First of all, the mobile interstitial ions will go up to the speck and discharge some of the electrons. This will take of the order of 10^{-5} seconds. Because of the slow rate of formation of new interstitial ions, this process soon comes to an end, certainly with silver chloride grains. A different mechanism now comes into play. The field set up by the charged and by now somewhat enlarged latent image (nucleus) will pull out some of the nearest silver ions from the lattice. . . . The holes so formed will be "repelled" by the charged silver specks and the holes near the latent image will be filled up by neighboring silver ions. Thus the process can be repeated. The holes will accu-

[7] MOTT, N. F., *Repts. Progress Phys.*, **6**, 186 (1939).
[8] VON ARDENNE, M., *Z. angew. Phot. Wiss. Tech.*, **2**, 14 (1940).
[9] HALL, C. E., AND A. L. SCHOEN, *J. Optical Soc. Am.*, **31**, 312 (1941).
[10] SHEPPARD, S. E., "Colloid-chemical aspects of photographic development," "Colloid Chemistry," Vol. 5, J. Alexander, ed., Reinhold Publishing Corporation, New York, **1944**.
[11] POLESITSKII, A. E., AND A. MURIN, *Doklady Akad. Nauk. S.S.S.R.*, **45**, 254-6 (1944); *Compt. rend. acad. sci. U.R.S.S.*, **45**, 236-40 (1944).
[12] BERG, W. F., *Trans. Faraday Soc.*, **39**, 126 (1943).

mulate on the parts of the crystal removed from the growing silver speck. . . . It will be seen that this process amounts to a dissolution of the crystal, all the moving of the silver ions, however, taking place in the solid, the bromide ions not being required to move through the lattice.

Berg emphasizes that the "rate of development cannot be determined by any considerations on the lattice and other energies involved" since the breakdown of the silver halide structure is an essential feature of development according to this scheme. It is not clear, however, why the breakdown of the crystal should occur more easily within the crystal than at the surface where the silver ions are more mobile and the halide ions could escape more easily.

Although Berg's modification avoids the specific objections which have been directed at the Gurney-Mott mechanism, the specific merits claimed for it do not appear to be genuine. For example, the induction period in development (*i. e.*, the time lapse before the appearance of detectable development) is identified with the interval during which the silver specks grow slowly at the rate at which fresh interstitial ions are formed. This explanation fails to account for the observed dependence of the induction period on the active charge of the developer ion[13] and for the practical absence of a genuine induction period in many cases. Neither does it explain why the induction period for a given developing agent can vary widely with the conditions under which the agent is used.

Several development mechanisms have been suggested by Russian scientists within the past few years. Anastasevich[14] has proposed a mechanism similar to that of Gurney and Mott and has worked out a semi-quantitative formulation for the rate of development under various conditions. Frank-Kamenetskii[15] and Bagdasar'yan[16] have shown that this mechanism is not in accord with the facts. Frank-Kamenetskii had previously proposed an electrode mechanism in which the over-all rate of development was determined by the rate of diffusion,[17] but this also is in contradiction to experimental results.[16] Bagdasar'yan, in his turn, suggests a mechanism which gives a much more satisfactory accounting of experimental results, but which, as we shall see, is not entirely free from objections.

Bagdasar'yan[16] considers first the reduction of silver ions on the surface of a silver particle and treats the process as that of the discharge of an ion at a static electrode. He assumes that the slowest stage in the reaction is the transition of the electron from the developer molecule or ion to the silver particle. Following Frumkin's formulation of the electrode process,[18] Bagdasar'yan derives the rate equation

$$d(\mathrm{Ag})/dt = k[\mathrm{Red}]e^{\alpha FV/RT}S \qquad (3)$$

where [Red] symbolizes the concentration of developing agent, α is a fraction, V is the potential drop at the border, silver/solution, and S is the area of the interface, silver/solution. V, however, can be expressed by the equation

$$V = V_0 + \frac{RT}{F}\log\,[\mathrm{Ag}^+] \qquad (4)$$

Hence, equation (3) may be rewritten:

$$d(\mathrm{Ag})/dt = k'[\mathrm{Red}][\mathrm{Ag}^+]^{\alpha}S \qquad (5)$$

Equation (5) is in complete agreement with the experimental data for the reduction of silver ions by hydroquinone,[19] and, in so far as dependence of rate upon silver ion concentration is concerned, it likewise is in agreement with data on the reduction of silver ions by hydroxylamine.[20] In each instance, the experimental value of α is approximately 2/3. The accord does not extend to the results obtained for the reaction between silver ions and *p*-phenylenediamine, however. Here,[21] the reaction rate is directly proportional to the silver ion concentration and proportional to a fractional power of the developer concentration. Moreover, the reaction shows a strong positive salt effect which is not predicted by Bagdasar'yan's treatment.

The suggested mechanism is readily extended to include the development of the silver halide grain. As before, the anode process (discharge of developer ions or molecules) could take place at any point along the silver/solution interface, but the cathode process (reduction of silver ions) now would be localized to some part of the silver surface very close to the silver/silver halide interface.

Evidently, the reduction of the silver ions cannot take place far from this border, since the drop in the concentration of the silver ions would be rapidly compensated by solution of the silver, owing to the current flow through the micro-element. The reduction of the silver ions can take place only along the border with the silver bromide, where the decrease in the silver ions will be compensated by the solution of silver bromide. It is very likely that the silver ions are reduced directly from the silver bromide crystal along a third boundary, since the concentration of the silver ions is much greater in the crystal than in the solution.

If the effective "concentration" of the silver ions available for reduction in direct development is determined by the concentration of bromide ions, the rate of development should be given by the equation

$$d(\mathrm{Ag})/dt = k_1[\mathrm{Red}][\mathrm{Br}^-]^{-\alpha}S \qquad (6)$$

(from Eq. 5). This equation is in agreement with data reported by Sheppard and Mees[22] for development by ferro-oxalate solutions, and by James[23] for development by hydroquinone. As we shall see later, however.

[13] JAMES, T. H., *J. Phys. Chem.*, **43**, 701 (1939); *J. Franklin Inst.*, **240**, 83 (1945).

[14] ANASTASEVICH, V. S., *Acta Physicochim. U.R.S.S.*, **16**, 296 (1942); *J. Tech. Phys. U.S.S.R.*, **14**, 467 (1944).

[15] FRANK-KAMENETSKII, D. A., *Acta Physicochim. U.R.S.S.*, **18**, 91 (1943).

[16] BAGDASAR'YAN, KH. S., *J. Phys. Chem. U.S.S.R.*, **17**, 336 (1943); *Acta Physicochim. U.R.S.S.*, **19**, 421 (1944).

[17] FRANK-KAMENETSKII, D. A., *J. Phys. Chem. U.S.S.R.*, **13**, 1403 (1939); *Acta Physicochim. U.R.S.S.*, **12**, 13 (1940).

[18] FRUMKIN, A., *Z. physik. Chem.*, **160**, 116 (1932); *Reports II of the Conference on Corrosion*, **1**, 5 (1940).

[19] JAMES, T. H., *J. Am. Chem. Soc.*, **61**, 648 (1939).

[20] JAMES, T. H., *ibid.*, **61**, 2379 (1939).

[21] JAMES, T. H., *J. Phys. Chem.*, **45**, 223 (1941).

[22] SHEPPARD, S. E., AND C. E. K. MEES, "Investigations on the Theory of the Photographic Process," Longmans, Green, and Company, London, 1907.

[23] JAMES, T. H., *J. Phys. Chem.*, **44**, 42 (1940).

Bagdasar'yan's mechanism, in common with the other electrode mechanisms, runs into difficulties in accounting for the dependence of the development rate upon the hydroquinone concentration.

Bagdasar'yan assumes that the same process takes place during the induction period as during the subsequent course of reduction of the individual grain, only a somewhat greater activation energy is required for reaction during the induction period. The induction period of the individual grain comes to an end when the properties of the silver nuclei become substantially those of massive silver.

A mechanism suggested by Huggins[2] is essentially an extension of his latent-image hypothesis but has some features suggestive of Berg's modification of the Gurney-Mott mechanism. The Huggins latent-image hypothesis proposes that photoelectrons trapped at the "sensitivity specks" induce a transition from the normal silver bromide *B1* crystal structure to the *B3* (or *B4*) structure in the immediate vicinity of the silver sulfide sensitivity speck. The localized region of *B3* (or *B4*) crystal structure, plus the excess electrons, constitutes the latent image.

> In the development process, extra electrons from the developer enter the surface of a *B3* region (more readily than of a *B1* region). After development has started, they may also enter at the silver surface. These electrons diffuse into the interior of the *B3* region, causing it to expand at the expense of the *B1* part of the crystal. . . . The extra electrons also produce migration of silver ions—especially in the *B3* structure and along the crystal surface, both where the underlying structure is *B3* and where it is *B1*—in such a way as to start a crystal of metallic silver on the surface. Further additions of migrating silver ions cause this silver crystal to grow. The ionic migrations. . . leave the bromide ions on the surfaces held only very loosely or not at all. . . . They therefore diffuse away into the solution. . . . In none of the assumed processes is any break-up and reformation of the crystals necessary: both the transition from *B1* to *B3* and from *B3* to metallic silver are continuous.

This mechanism is a highly specialized one. Energy calculations show that it is feasible for silver bromide. No detailed calculations have been made for silver chloride, but the indications are that a *B1* to *B3* shift is less likely in this material than in the bromide. The mechanism is not applicable at all to development of silver oxalate or silver iodide. As applied specifically to silver bromide, there is no real evidence either for or against the mechanism at the present time.

MECHANISMS INVOLVING DIRECT REACTION OF DEVELOPER AND SILVER ION

Piper, in 1908, had suggested that development is an "action of a catalytic nature, the latent image being the catalyzer," but he did not suggest a mechanism for the catalysis. Similarly, Volmer suggested that the silver of the latent image catalyzes the oxidation of the developing agent, but did not propose a mechanism.

The first mechanism of development which can be listed definitely under the heading of a triple interface reaction was proposed by Sheppard and Meyer.[24] Sheppard had shown[25] that colloidal silver markedly accelerates the formation of silver in a silver nitrate-sodium sulfite solution. Sheppard and Meyer suggested that in development, the reducing agent first is adsorbed by the silver halide surface, forming a complex with the silver ion, and that this complex decomposes in the presence of latent image silver to yield silver and oxidized developing agent.

Rabinovich and Peissakhovich[26] adopted another point of view, *i. e.*, that the basic cause of development lay in the adsorption of the developing agent by the latent-image nuclei and by the silver formed during subsequent development. This view was sharply criticized by Reinders,[27] but his criticism was based upon a complete misconception of the principles of adsorption catalysis and has no validity. However, the experimental evidence upon which Rabinovich based his mechanism, *i. e.*, evidence purporting to show adsorption of hydroquinone by colloidal silver directly, has not survived close examination. Perry, Ballard, and Sheppard demonstrated[28] that the experimental error in the work of Rabinovich and Peissakhovich was so large that their results do not actually demonstrate such adsorption. On the other hand, the results of Perry, Ballard, and Sheppard do not show conclusively that no adsorption of hydroquinone occurs.

If silver does adsorb hydroquinone at all, the adsorption must be weak, and there is good reason to doubt that it is of any importance to photographic development. Experiments on the silver-catalyzed reaction between hydroquinone and silver ions derived from a soluble silver salt show that the rate of the reaction is directly proportional to the hydroquinone concentration[19,21] and is proportional to a fractional power of the silver ion concentration. These results, as we have seen, are in accord with Bagdasar'yan's electrode mechanism. They also can be explained on the basis of adsorption catalysis, but adsorption of silver ions, not of hydroquinone. We can start from the normal assumption that the rate of reaction is proportional to the concentrations of the reactants. If one reactant is adsorbed prior to reaction, its significant concentration will be that in the *adsorbed* state, rather than in solution. Now, the concentration of a substance in the adsorbed state, C_a, often is related (to good approximation) to the concentration in solution, C_s, by the Freundlich equation

$$C_a = kC_s^{\alpha} \qquad (7)$$

where α is a fraction. Accordingly, if silver ion is adsorbed by silver but hydroquinone is not, the rate relation to be expected is

$$d(\mathrm{Ag})/dt = k'[\mathrm{Hq}][\mathrm{Ag}^+]_a = k''[\mathrm{Hq}][[\mathrm{Ag}^+]_s^{\alpha} \qquad (8)$$

[24] SHEPPARD, S. E., AND G. MEYER, *J. Am. Chem. Soc.*, **42**, 689 (1920).

[25] SHEPPARD, S. E., *Phot. J.*, **59**, 135 (1919).

[26] RABINOVICH, A. J., AND S. PEISSAKHOVICH, *Z. wiss. Phot.*, **33**, 94 (1934); A. J. RABINOVICH, *Trans. Faraday Soc.*, **34**, 920 (1938).

[27] REINDERS, W., *Trans. Faraday Soc.*, **34**, 936 (1938).

[28] PERRY, E. S., A. BALLARD, AND S. E. SHEPPARD, *J. Am. Chem. Soc.*, **63**, 2357 (1941).

and this is precisely the relation found experimentally. Thus, the kinetic evidence indicates adsorption of silver ions, but fails to indicate adsorption of the hydroquinone.

On the other hand, the rate of direct development of silver bromide grains[23] by hydroquinone in the pH region 8.0–8.9 varies as the 0.56 to 0.61 power of the hydroquinone concentration. This result (which is not in accord with Bagdasar'yan's formulation) indicates that hydroquinone in this instance is adsorbed prior to reaction. If silver were the effective adsorbent, there is no apparent reason why the kinetics of the reaction between hydroquinone and silver ions derived from a soluble salt do not show a similar dependence upon the hydroquinone concentration. The logical assumption, therefore, is that the hydroquinone in the development of silver bromide grains is adsorbed by the silver bromide or by the interface region, rather than by the silver. Similar results have been obtained with catechol as the developing agent.[29]

The silver-catalyzed reaction between certain other developing agents and silver ions derived from a soluble salt does show evidence of adsorption of the reducing agent by silver. Thus, the rate of reaction between silver ions and hydroxylamine shows the same dependence upon the silver ion concentration as that found for the hydroquinone reaction, but, unlike the latter, it shows a fractional power dependence upon the concentration of the reducing agent. The *p*-phenylenediamine reaction shows a fractional power dependence upon the concentration of the reducing agent and a *first power dependence* upon the concentration of the silver ion. In the *p*-phenylenediamine reaction, therefore, adsorption of the reducing agent probably is of major importance to the catalysis.

The most recently suggested form[30] of the triple-interface mechanism of development is a modification and elaboration of the Sheppard mechanism. Let us consider the reaction between a developing agent, say hydroquinone, and silver ions in solution in terms of the theory of "absolute rates." In the homogeneous, uncatalyzed reaction, an "activated complex" is formed between the two reaction partners. This complex can be represented by the symbol, $(D\epsilon \ldots Ag^+)^{\neq}$, and the total reaction by the scheme

$$D\epsilon + Ag^+ \rightleftharpoons (D\epsilon \ldots Ag^+)^{\neq} \rightarrow Ag + D \qquad (9)$$

The reaction must pass over an energy barrier corresponding to the activation energy. The rate of reaction is equal to the concentration of activated complex at the top of the barrier, multiplied by the frequency with which it passes over the barrier. The energy barrier is represented by the maximum of the solid curve in Figure 2.

In the catalyzed reaction, adsorption of the silver ion by the catalyst is the factor of major importance in permitting the reaction to proceed with a smaller activation energy than that of the homogeneous reaction. The catalyzed reaction can be treated as consisting of three reactants—the developing agent, the silver ion, and the adsorption site.[31] All three participate in the formation of the activated complex, which is then represented as $(D\epsilon \ldots Ag^+ \ldots S)^{\neq}$, where S is the adsorption site or sites. The activation energy for this reaction is appreciably smaller than that for the uncatalyzed reaction, and the reaction "path" is illustrated by the broken curve in Figure 2. The difference between the activation energy required for the catalyzed and the uncatalyzed reactions will equal the difference in potential energies of the two activated states, *i. e.*, the heat of adsorption of the activated complex minus the heat of adsorption of the reactants.

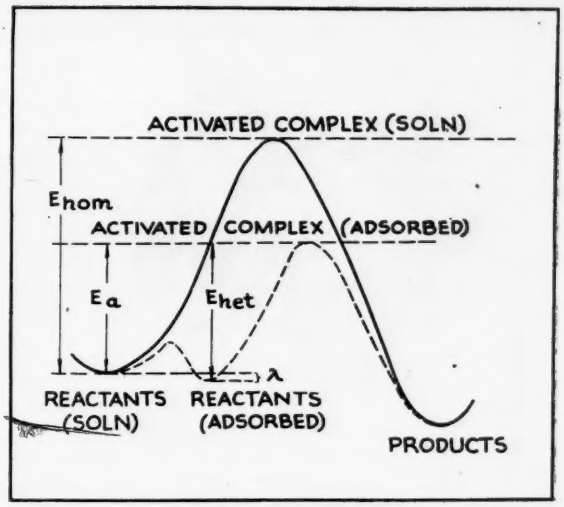

FIGURE 2

The rate of the homogeneous reaction is given by the equation,

$$v_{hom} = \frac{kT}{h} A' e^{-E_{hom}/RT} \qquad (10)$$

where k is the Boltzmann constant, h the Planck constant, and A' is a statistical factor involving the concentrations of the reactants and the "partition functions." Similarly, the rate of the heterogeneous, catalyzed reaction is given by

$$v_{het} = \frac{kT}{h} A'' e^{-E_{het}/RT} \qquad (11)$$

where the statistical factor A'' involves the concentrations of the reactants, including the adsorption site, and the partition function (which is not the same as the one of Eq. 10). The relative rates of the two reactions are then

$$\frac{v_{het}}{v_{hom}} = \frac{A''}{A'} e^{-\Delta E/RT} \qquad (12)$$

where ΔE is equal to $E_{hom} - E_{het}$. The value of ΔE taken

[29] JAMES, T. H., *J. Chem. Phys.*, **14**, (Sept., 1946).
[30] JAMES, T. H., AND G. KORNFELD, *Chem. Revs.*, **30**, 1 (1942); T. H. JAMES, *J. Chem. Phys.*, **11**, 338 (1943).
[31] LAIDLER, K. J., S. GLASSTONE, AND H. EYRING, *J. Chem. Phys.*, **8**, 667 (1940).

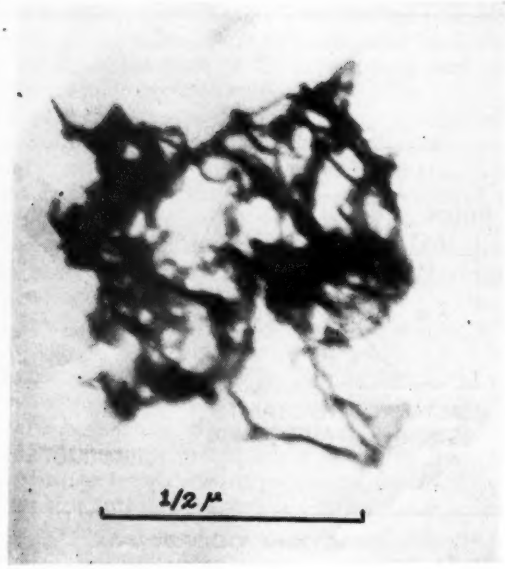

FIGURE 3. ELECTRON MICROGRAM OF PARTIALLY DEVELOPED GRAIN, SHOWING THE FILAMENTARY STRUCTURE OF THE SILVER AND TRIANGULAR ENLARGEMENTS OF SOME FILAMENTS

together with the relative values of A'' and A' determines whether the catalyzed reaction will proceed more rapidly than the uncatalyzed under any specific set of conditions. The heterogeneous, catalyzed reaction is, in general, favored by an increase in surface of the catalyst and by a decrease in the temperature at which the reaction is carried out.

The preceding scheme can be extended readily to cover direct development of a silver halide grain. The emphasis now is on the silver-silver halide interface as the most favorable spot for reaction. The silver ions at this interface are aleady subjected to adsorption forces and are probably displaced somewhat from their normal lattice positions. The activated complex is formed at this interface, and can be considered as the transition state between Sheppard's "adsorption complex" and the ultimate reaction products—silver and oxidized developer. The qualitative predictions (carried over from the treatment of the simpler reaction), that the catalyzed reaction is favored relatively by an increase in the surface of the catalyst and by a decrease in the reaction temperature, are in accord with the available evidence.[5,32,33] The first prediction is, of course, self-evident and would follow from any of the various mechanisms discussed previously.

Qualitatively, the proposed mechanism of development is in good accord with kinetic data on other scores. For example, the retarding action of bromide ions and of extraneous adsorbed material, such as gelatin and certain strongly adsorbed dyes, is readily accounted for. The concentration of the activated complex will depend upon the availability of the silver ions for combination with the developing agent as well as upon the effective concentration of the developing agent at the reaction site. Adsorption of the materials mentioned can decrease one or both. Quantitatively, no rigorous test for the proposed mechanism has been devised.

As we have seen, the dependence of the development rate upon a fractional power of the concentration of hydroquinone or catechol fits quite naturally into the triple-interface mechanism. On the other hand, this dependence constitutes perhaps the one bit of definite evidence against the electrode mechanisms in general. No explanation has been given on the basis of any form of the electrode mechanism, with the exception of the very special form proposed by Huggins. If adsorption of hydroquinone or catechol by the silver halide or the interface region must precede reaction, there is no apparent reason why the silver ion in the immediate vicinity of the adsorption site (indeed, probably the adsorption "site" itself) should not react with the developing agent rather than a silver ion at some distant point on the silver-silver halide interface.

THE FORMATION OF SILVER FILAMENTS IN DEVELOPMENT

We turn now to a brief consideration of a phenomenon which, at first glance, appeared to give strong support to an electrode mechanism of the type suggested by Gurney and Mott. This is the formation of silver filaments during the reduction of silver halide grains.[8,9] When partially developed grains are examined under the electron microscope, filaments are often observed which look as though they had been formed by squirting silver through an orifice. Moreover, silver extrusions can be obtained by heavily exposing a silver bromide grain to light or a beam of electrons. The Gurney-Mott mechanism very probably is followed in the formation of the latter and the silver extrusions are the result of an internal pressure forcing the photolytic silver specks out from the grain. These extrusions, however, do not resemble the development filaments in appearance. The former look much like warts on the grain surface and are thicker at the base than near the top. The latter generally take the form of long ribbons and frequently show angular enlargements in the plane of the ribbon. Many of these enlargements are flat, triangular crystals utterly unlike anything thus far observed in the photolysis of silver halide crystals. According to Jelley,[34] the filaments very probably are silver crystals growing chiefly in one dimension.

Some writers have sought to bolster the Gurney-Mott type of mechanism by drawing analogies between the filaments and hair-silver. However, the evidence is actually damaging to such analogies. Hair-silver has been obtained experimentally only by reduction of silver sulfide and silver chloride with hydrogen. The hair-silver is obtained most readily by reducing solid silver sulfide in the temperature range of 300 to 600°C. The mobility of silver ions in the silver sulfide is quite

[32] JAMES, T. H., *J. Am. Chem. Soc.*, **62**, 1649 (1940).
[33] SHIBERSTOFF, V. I., AND YU. I. BUKIN, *Kino-Photo-Chem. Ind.*, **1**, 101 (1932).
[34] JELLEY, E. E., *J. Phot. Soc. Am.*, **8**, 283 (1942).

high at such temperatures, and it seems plausible, as Kohlschütter assumes,[35] that at least a portion of the silver making up the fibers comes from within the silver sulfide particle. The situation, however, is not comparable to that of reduction of silver bromide. At the temperatures involved, thermal dissociation of silver sulfide is occurring according to the general equation

$$Ag_2S \rightarrow S + 2\epsilon + 2Ag^+$$

so that no difficulty is encountered here in explaining what happens to the sulfide ions.

The formation of hair-silver by reduction of silver chloride lends no support to the Gurney-Mott type of mechanism. Hair-silver has not been obtained from silver chloride in the *solid state*. Kohlschütter obtained it only when the temperature of the silver chloride was raised to 550°C., and this is 95° *above* the melting point of the salt. Obviously, extrusion of silver by pressure from within cannot be invoked in this instance.

Jelley's observations on the form of the development filaments suggest that the filament formation is a crystallization phenomenon. Evidence bearing on the mechanism of this crystallization process has been examined elsewhere.[36]

[35] KOHLSCHÜTTER, H. W., *Z. Elektrochem.*, **38**, 345 (1932).

[36] JAMES, T. H., *J. Chem. Phys.*, **11**, 338 (1943).

A Versatile Microscope Hot Stage

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THE ease with which thermal behavior of substances may be studied microscopically has given rise to an extensive literature (*1, 2, 3*) describing the construction and use of hot and cold stages for the microscope. A number of the hot stage designs are specifically for the determination of melting points, and the authors claim accuracy and control comparable to or better than that attained by the customary heating-bath methods. The advantages of employing a microscope for melting point determinations, especially in conjunction with the use of polarized light, are summarized by Chamot and Mason (*1*). Such points in favor, as the use of smaller quantities of material, the increased ease in recognizing and distinguishing accompanying thermal behavior, the readiness with which impurities are detected, and the wider range of temperatures available, argue for a more extensive use of the microscopic method.

The majority of the hot stage designs previously proposed, while possessing many desirable features, have several disadvantages in common. In the first place, due to a complete enclosure of the sample within the stage walls, there is no provision for removing or introducing samples after the stage is heated. Secondly, for the same reason, there are no means of examining any but a single portion of the field during heating. This is a distinct disadvantage, since the most significant portions of a melt are not necessarily in the field of the objective at the outset. Lastly, the cover glasses upon which samples are usually placed are less convenient to manipulate than slides. A need on the part of the author for a more versatile hot stage has prompted a design which eliminates these disadvantages while retaining earlier desirable features.

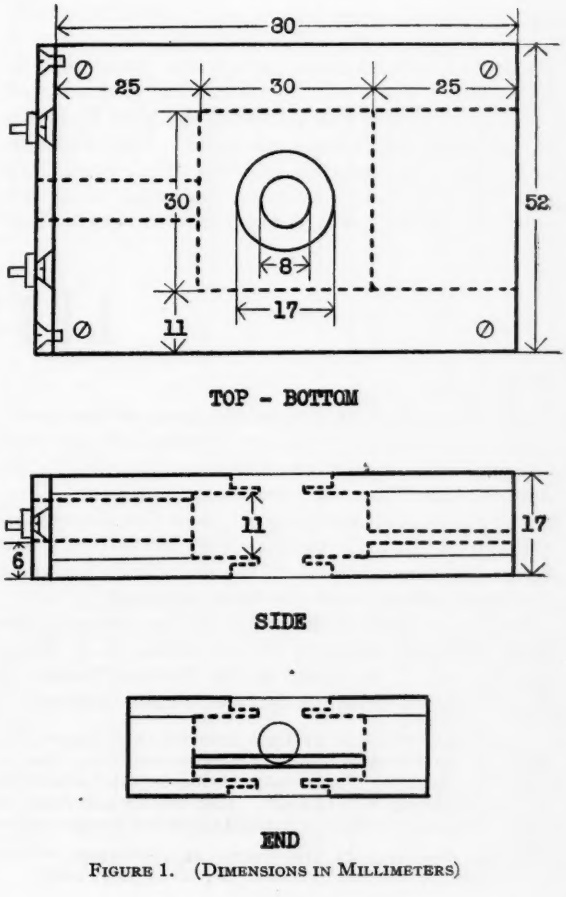

FIGURE 1. (DIMENSIONS IN MILLIMETERS)

The present design utilizes an electrically heated metal block provided with a square heating chamber in the center large enough to accommodate an ordinary slide. Visual access to the chamber is provided by glass windows on the top and bottom of the stage. A horizontal hole in one end of the stage permits the insertion of a thermometer into the heating chamber, and a horizontal slit in the other end provides for introduction, withdrawal, or manipulation of a slide in the chamber at any time during the operation of the stage. The sample being studied is contained between a slide and cover glass.

The stage is constructed entirely of brass according to the dimensions (millimeters) of Figure 1. The center block, containing the heating chamber, consists of a single piece, with the exception of a thin bridge, 2 mm. thick, screwed into place under the 2-mm. slide slit. Holes are drilled in the 3-mm. top and bottom pieces and recessed around the circumference for the insertion of glass windows. The top and bottom are then screwed to the center block, forming a single unit. A piece of one-eighth inch Transite board, having the dimensions of one end of the unit and drilled with a 7-mm. hole for insertion of the thermometer, is screwed to the thermometer end of the stage. The Transite is drilled and countersunk for the attachment of binding post screws and serves both as a nonconducting anchor for the binding posts and as insulation for one end of the stage. Sixteen-millimeter circular glass windows (polarimeter windows) are secured in their recessed retainers in the top and bottom of the stage by means of a pulp made of asbestos and water. The entire unit is wrapped with damp asbestos tape, dried, wound with ten feet of No. 28 Chromel A resistance wire, then covered with six layers of damp asbestos tape. After drying, the slide slit is readily cut open with a razor. Before assembly the walls of the heating chamber are blackened with lampblack to increase internal radiation (*1*). The assembled unit is advantageously painted with a mixture of aluminum bronze and sodium silicate solution. The present stage was designed for use with an inexpensive student microscope and is attached by unscrewing the stage of the latter and bolting the hot stage in position in its place.

The heating unit is operated from a 5-ampere Variac transformer, and it is convenient to calibrate the Variac settings in terms of the stage temperatures. A temperature of 350° was attained with the Variac set at 60. The thermometer is inserted into the heating chamber to the point where it just begins to appear in the field. The sample to be examined is placed on one end of a slide, pressed under a cover glass, and cautiously inserted into the slit so as not to cause the cover glass to slip. By employing both ends, a single slide suffices for two observations. Microscopically determined melting points are generally low due to the ease with which incipient fusion is observed (*1*), and it is therefore desirable to calibrate the stage by means of a series of pure samples of known melting point.

The author is grateful to Mr. A. Jansse for aid in the construction of the hot stage described.

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LETTERS

To the Editor:

In connection with the development of the atomic bomb, elements 93 to 96 were synthesized and their chemical and physical properties more thoroughly examined than those of some natural elements. Seaborg and his coworkers have proposed that elements 90 through 96 constitute the first seven elements of a new series of elements, similar to the rare earths, for which the name *actinide series* has been suggested.[1]

Further evidence of the validity of this conclusion has been published recently. C. C. Kiess, C. J. Humphreys, and D. D. Laun[2] of the National Bureau of Standards said about the first spectrum of uranium:

> From the spectroscopic evidence presented in this paper, it is clear that uranium is an element of the rare-earth type; that, in fact, it is a homolog of neodymium, being the third member of a series beginning with thorium. That the seventh period of elements in the periodic system should encompass a second group of rare earths similar to the group in the sixth period was foreseen more than 20 years ago by Bohr.[3]
>
> Some investigators, notably Villar, have suggested Th (90), others U (92), and still others, including Starke, the transuranium element 93 as the first in which a 5f electron appears. In each instance the basis of opinion was evidence derived from chemical behavior, nuclear activity, behavior of ions in crystals and solutions, etc. The testimony of the spectroscope, however, in selecting thorium for this role is unique and decisive, and it is supported by the recent spatial classifications of the elements by Djownkorsky and Karis[4] and by Talpain[5] which array thorium, protoactinium, and uranium as the chemical homologs of cerium, praseodymium, and neodymium.

I should like to suggest that you include a copy of the enclosed periodic table as a Christmas present to your readers (see page 603).

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[1] *Chem. Eng. News*, **23**, 2190 (1945); **24**, 1197 (1946).
[2] *J. Research Nat. Bur. Standards*, **37**, 57–72 (July, 1946).
[3] *Nature*, **112**, 43 (1923).
[4] *J. phys. radium*, (8) **5**, 53 (1944).
[5] *Ibid.*, (8) **6**, 176 (1945).

PERIODIC CLASSIFICATION OF THE ELEMENTS

The atomic numbers are placed above the symbols of the elements; the atomic weights below the symbols.

Group	Ia	IIa	IIIa	IVa	Va	VIa	VIIa	VIII	Ib	IIb	IIIb	IVb	Vb	VIb	VIIb	0
Type	R_2O RH	RO RH_2	R_2O_3	RO_2	R_2O_5	RO_3	R_2O_7	RO_4	R_2O	RO	R_2O_3	RO_2 H_4R	R_2O_5 H_3R	RO_3 H_2R	R_2O_7 HR	Inert Gases
Period 1 2 Elements	**1** H 1.0080															**2** 2 He 4.003
Period 2 8 Elements	**3** Li 6.940	**4** Be 9.02									**5** B 10.82	**6** C 12.010	**7** N 14.008	**8** O 16.0000	**9** F 19.00	**10** 2 8 Ne 20.183
Period 3 8 Elements	**11** Na 22.997	**12** Mg 24.32									**13** Al 26.97	**14** Si 28.06	**15** P 30.98	**16** S 32.06	**17** Cl 35.457	**18** 2 8 8 A 39.944
Period 4 18 Elements	**19** K 39.096	**20** Ca 40.08	**21** Sc 45.10	**22** Ti 47.90	**23** V 50.95	**24** Cr 52.01	**25** Mn 54.93	**26** Fe 55.85	**27** Co 58.94	**28** Ni 58.69	**29** Cu 63.57	**30** Zn 65.38	**31** Ga 69.72	**32** Ge 72.60	**33** As 74.91	**34** Se 78.96	**35** Br 79.916	**36** 2 8 18 8 Kr 83.7
Period 5 18 Elements	**37** Rb 85.48	**38** Sr 87.63	**39** Y 88.92	**40** Zr 91.22	**41** Cb 92.91	**42** Mo 95.95	**43**	**44** Ru 101.7	**45** Rh 102.91	**46** Pd 106.7	**47** Ag 107.880	**48** Cd 112.41	**49** In 114.76	**50** Sn 118.70	**51** Sb 121.76	**52** Te 127.61	**53** I 126.92	**54** 2 8 18 18 8 Xe 131.3
Period 6 32 Elements	**55** Cs 132.91	**56** Ba 137.36	**57** *58–71 La 138.92	**72** Hf 178.6	**73** Ta 180.88	**74** W 183.92	**75** Re 186.31	**76** Os 190.2	**77** Ir 193.1	**78** Pt 195.23	**79** Au 197.2	**80** Hg 200.61	**81** Tl 204.39	**82** Pb 207.21	**83** Bi 209.00	**84** Po (210)	**85**	**86** 2 8 18 32 18 8 Rn (222)
Period 7	**87**	**88** Ra 226.05	**89** **90–96 Ac (227)															

Transitional Elements

*Rare Earth Elements (Lanthanide Series) Group IIIa	**58** Ce 140.13	**59** Pr 140.92	**60** Nd 144.27	**61**	**62** Sa 150.43	**63** Eu 152.0	**64** Gd 156.9	**65** Tb 159.2	**66** Dy 162.46	**67** Ho 164.94	**68** Er 167.2	**69** Tm 169.4	**70** Yb 173.04	**71** Lu 174.99

**Actinide series Group IIIa	**90** Th 232.12	**91** Pa 231	**92** U 238.07	**93** Np (238)	**94** Pu (238)	**95** Am (242)	**96** Cm (243)

Simple Devices for Measuring Out Small Quantities of Liquid[1]

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INTRODUCTION

ALTHOUGH microchemical operations constantly require the use of small droplets of liquid, books on microchemistry tell surprisingly little as to how droplets[2] of closely predictable volume can be formed. Quantitative data on this subject must now and then be useful even in qualitative work, and if they were generally available they might cause microchemists to try more frequently for results that are at least roughly quantitative.

The size of a droplet formed with most of the implements to be described—the one exception being the sleeved pipet, which is less accurate than the others—depends on surface tension and will differ for different liquids. It will also be affected in minor degree by temperature and pressure. The results given here are based on experiments with tap water under ordinary room conditions. Others may find it worth while to plot curves for a few of the reagents that they employ most frequently, but in most cases the curves in Figure 2 will perhaps give a sufficiently close approximation.

Three of the implements here described—the open loop, the contact pipet, and the curved form of the plane-ended rod—were discussed in an earlier paper (*2*), which, however, contained no quantitative data regarding sizes of droplets. All three implements have been so designed as to be easily manipulated. Each has a substantial handle which slopes at about 30° when the implement is being used; the tip of the implement should therefore make that same angle with the axis of the handle, so as to lie horizontal when a droplet is being deposited (see Figure 1C). Short (*3*, p. 52) has pointed out the advantage of this "golf-club" profile for a loop.

LOOPS

Construction. A loop may be open or closed (see Figure 1, A and B). There is an important difference in behavior, which does not seem to be generally recognized, between these two kinds of loops. An open loop, broken by a gap at least as wide as the thickness of the wire, will release virtually all of its charge when the liquid is brought in contact with a slide, while a closed loop—or one having a very small gap—will retain a lens of liquid having about a third the original volume of the charge. Before putting a loop into regular use, the user should make sure that it behaves consistently in this respect.

Loops for depositing reagents are usually made of platinum wire, but platiniridium (Ir 15 per cent) has the advantage of being stiffer.

An open loop is preferable to a closed one when quantitative results are desired; a very small closed loop may be found useful, however, in depositing a droplet that must be confined to a small area, as is often desirable in etching a polished section of ore. The easiest way to make such a loop is to double a piece of fine wire around a No. 12 needle, or an even smaller piece of steel wire, and twist it snug. The loop should then be made as flat as possible by squeezing or gentle hammmering, and further dealt with as described below.

Open loops of the rather small sizes that are most useful can be neatly made as follows (see Figure 1B). A needle of the right diameter to serve as a core is driven into a piece of fine-grained wood and another needle, the smaller the better, driven close alongside it. A piece of annealed platiniridium wire, about 0.2 to 0.5 mm. thick and 15 to 20 mm. long, depending on the size of the loop, is wound around the core, crossing itself between the two needles, and is pulled snug by means of pliers or a needle holder. The loop is then laid on a smooth surface, preferably of marble, and the fag-end cut off with a knife or a small straight dental chisel. It is finally given the profile shown in Figure 1C. Smooth-faced pliers with parallel jaws are useful for this purpose, for flattening the ring, and for bringing the shank in line with the center of the loop.

The shank of the loop is fused into the drawn-out end of a piece of thin-walled glass tubing, preferably Pyrex (Figure 1C). To avoid cracking the glass when the loop is fire-cleaned, the tip of the handle should be warmed before bringing the wire to incandescence. Cracking can also be avoided by giving the loop a long shank, but a short shank economizes platinum and is less easily bent than a long one.

Charging. The amount of liquid that can be taken up in a loop of given size depends, to a degree that I have never seen adequately described, upon the way in which the loop is charged. If a closed loop is held vertical, dipped, and withdrawn slowly, as recommended by some writers (*1*, p. 42), it will contain a thin bi-concave lens of liquid. If a closed loop, held vertical, is withdrawn quickly, it will contain a sheet of liquid almost uniform in thickness. An open loop withdrawn when vertical will bring up no charge at all. If a closed loop is dipped while horizontal and raised slowly, it

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[2] "Droplet" is here used to designate a lens of liquid, lying on a flat surface or held in a loop.

will contain a thin bi-convex lens of liquid, and so, usually, will an open loop, although it may sometimes fail to hold a charge. But if a loop, whether closed or open, provided its inside diameter is not more than about 3.5 mm., is held horizontal and withdrawn quickly, it will hold a lens of liquid that is strongly bi-convex (Figure 1D).

The volume of the charge taken up by this quick horizontal dip is remarkably constant for a loop of given outside diameter and is little affected by minor variations of technique. The lifting motion may be of small amplitude or large, provided it be not violent; a violent jerk will cause much of the liquid to fall back. It matters little whether the loop is pushed through the surface film or only depressed until the film is visibly dented. The most uniform results, however, seem to be obtained by barely breaking the film, and then giving the hand an upward twitch without moving the arm. This motion need not raise the loop more than 1 cm. from the surface of the liquid, so that a charge can be dipped in this way from a small reagent bottle that is not too small in the throat.

Measuring the Charge. Others will probably wish to verify the results here given, and they may find it worth while to calibrate their own implements, for, owing to slight irregularities of form, the full charges of two loops having the same outside diameter may differ appreciably.

Charges can most easily be measured by means of a hand-made capillary pipet. As such a pipet will generally flare upward from the tip, it should be calibrated by weighing, once empty and at least three times more when filled to heights of, say, 25, 50, and 100 mm. The data thus obtained will give a calibration curve, from which the volume corresponding to any height of column can be read off by placing the pipet with the lower meniscus at the zero point. Several charges may be taken up successively without emptying the pipet, and if the increment due to each charge is noted, the range of variation as well as the average volume can be determined.

Care must be taken to see that the pipet imbibes each droplet quickly and completely; otherwise large negative errors may be caused by evaporation or by failure to get all of the liquid out of the loop. Errors may also be caused by air bubbles between charges. Unless due precautions are taken, the charges may appear to be less constant than they really are. Care is especially needful in calibrating small loops. As a result of numerous careful measurements, I have concluded that the volume of any single charge taken by a given loop in the manner advised above will rarely differ from the average by more than five per cent.

Results of Measurements. The relation between the outer diameter of a loop and the charge it brings up when given a quick horizontal dip is shown by curve A, Figure 2. This relation has been determined with much care because the quick horizontal dip gives a loop the largest closely predictable charge that it can hold. A larger charge can indeed be fed in with a rod, as suggested by Short (*3*, p. 54), but its volume cannot be so accurately controlled. Further advantages of taking a full charge, as the charge taken by quick horizontal dipping may be called, will appear when the mode of depositing the liquid is described. Curves have also been drawn to represent the charges taken up by slow-horizontal, quick-vertical, and slow-vertical dipping (Figure 2B, C, and D). Charges taken by these methods are less constant than the full charges and were measured with less care, but these curves will serve to show how great a difference the mode of dipping can make. For a 3-mm. loop the full charge is about nine times as much as the charge taken by a slow vertical dip, and about 50 times this minimal charge can be fed in with a rod.

Outer diameter is given, rather than inner diameter as is customary, because by so doing the effect of differences in thickness of wire is almost completely eliminated (see Figure 1D). The full charge, as determined experimentally, of water for a loop 2 mm. in

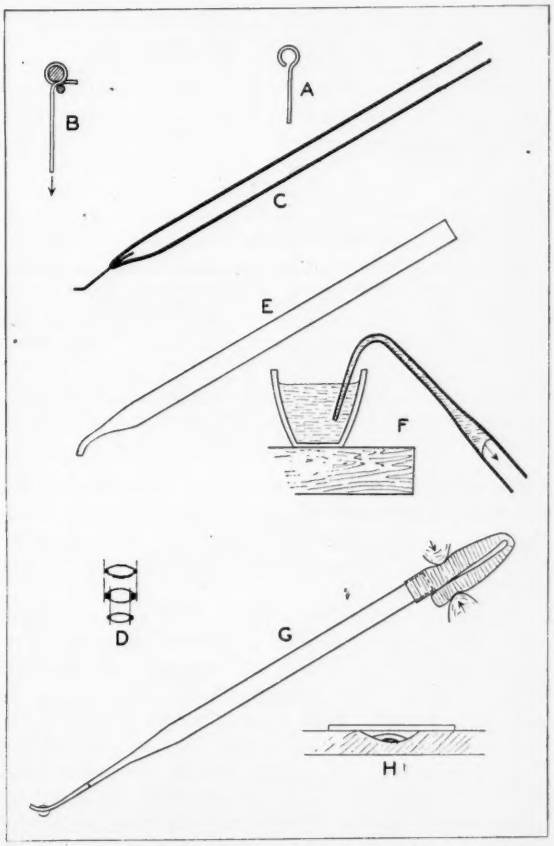

FIGURE 1.—IMPLEMENTS FOR MEASURING OUT SMALL QUANTITIES OF LIQUID (HALF NATURAL SIZE)

A, open loop; B, method of forming an open loop; C, section of loop with glass handle; D, section of charges in loops with equal outer diameters and equal inner diameters; E, profile showing form of curved plane-ended rod and of contact pipet; F, capillary siphon; G, sleeved pipet with charge partly expelled; H, section of droplet covered with oil.

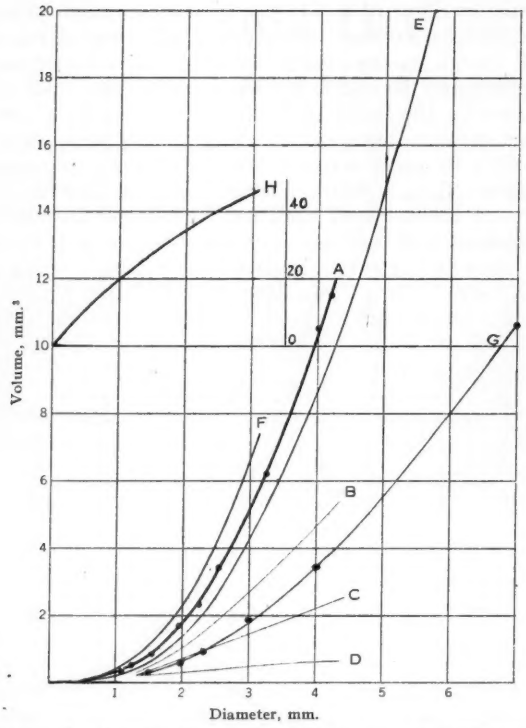

FIGURE 2.—CURVES SHOWING VOLUMES AND DIAMETERS OF DROPLETS OF WATER DEPOSITED IN VARIOUS WAYS

A, full charge of loop, taken by quick horizontal dip, plotted against outer diameter of loop; B, charge taken in loop by slow horizontal dip; C, same taken by quick vertical dip; D, same taken by slow vertical dip; E, full charge of plane-ended rod, plotted against diameter of sole; F, first droplet deposited from normal charge of contact pipet, plotted against outer diameter of tip; G, volume (+ 10 mm.³) of droplet laid down gently on a clean slide, plotted against diameter; H, volume of drop falling from dropper, plotted against outer diameter of tip. Interval between horizontal lines represents 20 mm.³

outer diameter is about 1.8 mm.³, regardless of the thickness of the wire, whereas a loop of 2-mm. inside diameter, made of thick wire, may take up more water than a loop of 2.5-mm. inside diameter made of thin wire (see Figure 1D).

Depositing Liquid from a Loop. The minimal charge taken up in a closed loop by slow vertical dipping is released only with difficulty, by tapping, and therefore tends to form a thin ragged splotch that cannot be accurately placed. With a fully charged open loop, the case is happily different, as may best be seen when a droplet is placed on a grain under the microscope. When the loop comes close to the grain, the latter becomes clearly visible, with its magnification augmented by the liquid lens. The downward movement of the hand is then instinctively retarded, and the loop centered on the grain. A moment later, the bottom of the liquid lens comes in contact with the slide, whereupon the liquid is quietly and wholly, or almost wholly, released. At most, a part of the wire will be wetted by a negligible amount of liquid, so that the deposit is virtually equal to the charge. As a droplet thus gently laid down will be nearly circular and will have the highest convexity that it can maintain, it will last longer before drying up than a thin irregular droplet.

What has been said in the last paragraph is equally true of a closed loop, except that a closed loop discharged in the manner described will retain a thin biconvex lens of liquid. The proportion of the charge that is deposited will largely depend upon the condition of the slide and the way in which the loop is handled. In general, however, the amount of liquid deposited by a closed loop on a clean slide is somewhere near two-thirds of the charge.

PLANE-ENDED RODS

It is not possible to deposit a droplet of closely predictable size with an ordinary stirring rod or with a rod drawn out to a point. Such a droplet can be deposited, however, with a rod terminated by a plane surface, which may be called a sole. A rod to be used for taking large droplets from a bottle may be left straight and of its original diameter. One that is to be used for placing small droplets accurately under the microscope should be drawn out at the end and given the profile shown in Figure 1E. A plane sole, even on a tip less than 1 mm. in diameter, may be ground with a lap or by hand on fine sand- or carborundum paper, the rod being held as it is when in use, but the sole will then be more or less rounded. Fire-polishing makes for cleanliness, but it reduces the effective diameter to an uncertain extent.

Instead of a glass rod with a very small tip, one may use a piece of platiniridium wire fused into a glass tube with a sole ground on the end.

To give a plane-ended rod its full charge, the sole, held horizontal, is barely immersed and the rod raised quickly. The sole will then be capped with a button of liquid, which usually stays in place when the rod is turned end for end. Curve E, Figure 2, gives approximately the relation between diameter of sole and volume of charge, which is measured by taking it from the rod with a calibrated capillary pipet. Nearly all of the charge is entirely released if the liquid is brought in contact with a slide while the sole is nearly vertical, and the rod then slowly raised. Volume of deposit is not quite so closely predictable as with open loops, but will not often differ by more than 10 per cent from that given by the curves. If a droplet is to be carefully placed with a curved rod, the sole had best be held horizontal while the liquid is deposited. When that is done, only about two-thirds of the charge is released.

CONTACT PIPETS

Depositing Liquids. A charged pipet having a profile similar to that of the rod shown in Figure 1E will deposit a droplet when the tip is brought into close contact with a clean slide and quickly raised. A sole as little as 1 mm. across can be ground on very fine sandpaper or carborundum paper. It must not be fire-polished if the pipet is to be used for depositing liquids.

What may be called the normal charge is imbibed by

capillary action when the tip is barely immersed and the tube held at an angle of 30°. If the tip is then repeatedly brought into close contact with a clean slide a series of droplets, each a little smaller than the last, will be deposited until the pipet is empty. Curve F in Figure 2 gives approximately the relation between the first droplet deposited from the normal charge and the diameter of the tip; with contact pipets as with loops the size of the droplets is determined by outer diameter.

If the pipet is considerably overcharged, which can be done by suction, the droplet formed on contact will grow until the tip is withdrawn. A droplet that is roughly of a predetermined volume, as estimated from its diameter, can thus be formed; but this method cannot give accurate results, for it is impossible to be sure of lifting the tip at the right instant, and the flow cannot be quickly arrested by putting the finger over the butt end of the pipet. As a depositor of droplets, the contact pipet is less adapted to quantitative work than the other implements here described.

Transferring Liquids. A contact pipet, preferably one with a slender tip, can serve as a "drinker" for taking up droplets. If the tip is very small, it had best be broken off squarely on a scratch made with a chip of quartz, then cautiously fire-polished to the least possible extent. A droplet can, of course, be taken up with a straight pipet or a bit of capillary tubing, but the curved pipet is more easily manipulated. If the liquid is to be redeposited, it can be expelled by gentle blowing or by means of the rubber sleeve described below. When the pipet is held with the bend pointing down, the expelled liquid will hang from the bend and will be released when brought in contact with a slide (see Figure 1G).

A curved pipet with the tip extending well beyond the bend will act as a capillary siphon (see Figure 1F). Capillary action pulls the liquid over the bend, and the liquid will then fill the tube and flow from the butt end as long as the tip is kept immersed.

SLEEVED PIPETS

Liquid can be expelled from a pipet by means of a rubber sleeve slipped over the butt end. The tube is folded as shown in Figure 1G and gently pinched between thumb and finger. Air pressure can be more delicately controlled in this way than with a bulb or with a tube stopped at the end. A droplet can thus be divided into roughly equal portions as little as 0.5 mm.^{3} in volume.

The sleeved pipet, unlike the other implements here considered, gives results that are independent of surface tension, but it will not form droplets of closely predictable size.

DROPPERS

The size of a drop falling from a tube depends on the diameter of the tip. The relation, as approximately determined by trial with droppers 0.19 to 3.0 mm. in tip diameter, is shown in curve H, Figure 2. The volume of the drop is nearly the same whether the drop is allowed to grow until detached by its own weight or is made to fall by means of a bulb.

The droppers put in small reagent bottles commonly have a tip diameter of about 3 mm. and give drops about 45 mm.^{3} in volume, which is far too large for direct use in a truly microchemical operation. The tip of a medicine dropper can easily be drawn down, however, to a diameter of less than 0.5 mm. without being over-long; a tip 0.45 in diameter gives drops of about 10 mm.^{3} In order to get an abrupt decrease in diameter, the drawing-out process should be repeated once or twice. The tip can then be broken square across on a scratch made with a chip of quartz. Manufacturers probably could make droppers 1 mm. in tip diameter, which would form drops about 20 mm.^{3} in volume, and if droppers are put in reagent bottles for microchemical use, there is no apparent reason for having them larger.

The only thing a dropper will do that the other implements here described cannot is to place drops on a wax plate, as is done in making certain colorimetric tests.

AUGMENTING OR DIMINISHING A DROPLET

A droplet, whether it be on a slide, on the up-ended sole of a rod, or in a loop, can be augmented by a fairly definite amount , or diminished by an amount that may be much less definite.

To add liquid, a charged loop or the sole of a charged rod, held nearly vertical, is inserted into the droplet and slowly withdrawn. To subtract liquid, the quick horizontal-dip method is used. A small rod or loop will take up nearly its full charge from a sufficiently large droplet, but when the volume of the source droplet decreases below a certain point, the volume of the charge that can be dipped from it with a given implement will rapidly diminish. A droplet therefore cannot all be divided into equal fractions by this method, but several nearly equal portions can be taken out for use in different tests.

RELATION OF DIAMETER TO VOLUME

Diameter does not afford a very reliable measure of volume because it is affected by the condition of the surface on which the liquid is deposited. A bead of liquid dropped on wax will have overhanging sides; one placed on heavily frosted glass with a fully charged loop will have about the same diameter as the loop; and one placed on an ordinary slide will spread beyond the diameter of the loop—more if the slide is clean and bright than if it is greasy or dull.

Curve G in Figure 2 gives approximately the relation, as determined by experiment, of diameter to volume for water laid down on clean, bright slides. This curve could be used for estimating roughly within how small an area a droplet laid down with a given implement can be confined or for judging what implement had best be used for laying down a droplet that must be confined within a given area. These questions, however, are most likely to be asked with regard to very

small areas, such as may be occupied by a certain mineral in a polished section. As no great accuracy can be claimed for the part of the curve relating to diameters less than 1 mm. and as something must be allowed for the tremor of the hand, the answers may best be obtained by experiment, the curves being used only as a first approximation. It is difficult to confine a droplet to a circle less than 0.5 mm. in diameter; and such a tiny droplet will usually evaporate within less than a minute, unless its life is prolonged by the method now to be described.

COVERING A DROPLET

If a very small droplet placed on a polished section seems likely to evaporate so quickly that it does not afford a fair test of an etch reaction, its life can be prolonged for several minutes if it is immediately covered, by means of a loop, with a large droplet of xylene. The droplet of reagent will not begin to evaporate until its top is exposed by the dwindling of the droplet of xylene.

A droplet of aqueous liquid can be preserved indefinitely by placing it on a hollow-ground slide or a spot-plate and covering it with mineral oil by means of a loop or a rod (see Figure 1H). The droplet is less likely to be pushed aside by the oil if it is first covered with xylene. This procedure may be useful when time is wanted for allowing a fine precipitate, such as ammonium phosphomolybdate, to settle, or for dissolving material that is slowly soluble at moderate temperature. The preparation can be warmed for a little while on the steam bath, but if it becomes too warm, bubbles of vapor will be formed and the droplet will be replaced by an oily froth.

When the experiment is concluded, the oil is easily removed by repeated washings with xylene. The xylene may be added with a loop or a rod and the mixture drawn off with a contact pipet or a capillary siphon.

ACKNOWLEDGMENT

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Nonsilica Glass Systems

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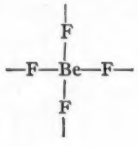
Bausch & Lomb Optical Company, Rochester, New York

FROM the technological viewpoint, three systems other than silicate systems deserve intensive study. Fluoride, phosphate, and borate glasses resemble silicate glasses in general principles of constitution but differ from each other in important chemical properties.

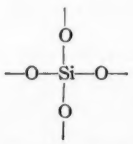
The common constitutional principle is the interlinkage of small strong cations with four surrounding highly polarized and relatively large anions. The total charge of these four anions is about twice that of the central cation. The arrangement is random, having been preserved in this randomness on cooling from the liquid state because the interlinkages are strong enough to prevent organization to the symmetry of the otherwise similarly constituted crystals of the family.

FLUORIDE GLASSES

In fluoride glasses the anion is the monovalent fluorine ion, while in oxide glasses, silicates, phosphates, and borates, it is the divalent oxygen ion. In the single compound glass, BeF_2, beryllium is divalent.



Four monovalent fluorine ions surround it to form groups interlinked at each of the four fluorines. A polymer $(BeF_2)_n$ results which is entirely analogous to, although more weakly bound together than, the corresponding



groups forming the polymer $(SiO_2)_n$, well known in the symmetrical modifications quartz, tridymite, and cristobalite and the randomly arranged silica glass. Because of the weaker bonds BeF_2 is distinguished from silica by high solubility, low viscosity, low density, low optical refraction, and absorption. It could be the base glass of X-ray windows and of low dispersion optics, but its extensive application is barred by its hygroscopic nature.

The combination of fluoride and oxide structure—historically preceding the discovery of the pure fluoride glass—is a compromise of high technological interest. It is limited by the volatility of fluorine from oxide-glass melts and the tendency of fluorides to crystallize from such melts. Not more than about 20 per cent can be,

and not more than 10 per cent of oxygen usually is, replaced by fluorine. The presence of those alkaline earths and divalent ions whose fluorides precipitate easily from silicates is avoided. The so-called opal glasses thus formed by the precipitation of fluorides are not a subject of this paper. Potassium, aluminum, or both are used to combine fluorine in clear silicate glasses. The applications of fluoride-oxide glasses are low-viscosity glasses, enamels, vitreous colors, and glasses transparent to ultraviolet light. Glasses containing fluorine are also used where iron-bearing raw materials must be made into glass free from the green color that iron gives to regular silicate types. The iron ion loses its visible color if surrounding oxygens are replaced by F. Fluorine is also used in fluor-crowns—that is, optical glass of low index of refraction and relatively low dispersion. These glasses were formerly imported but they are now available in the U. S.

PHOSPHATE GLASSES

In phosphate glasses the corresponding building unit is a PO_4 group. However, P^{5+} being pentavalent, more oxygen is available than required in a network where all oxygens are shared between $P_5{}^{5+}$. The polymer $(P_2O_5)_n$ thus formed is looser than $(SiO_2)_n$ but resembles $(Na_2Si_2O_5)_n$, the sodium disilicate glass with the same ratio of oxygen to silicon. The vitreous polymer $(P_2O_5)_n$ is more stable than crystalline P_2O_5, which has a higher vapor pressure and is only slightly polymerized. Glasses derived from these simple glasses, however, are not at all similar in chemical respect, because the same anionic structure in a silicate glass requires electropositive ions like sodium.

It is of high theoretical and practical interest that the combination (or alternation) of trivalent Al^{3+} and pentavalent P^{5+} leads to glassy $AlPO_4$ which is not only formally but also constitutionally equivalent to $SiSiO_4$ (SiO_2).

Glasses between P_2O_5 and $AlPO_4$, such as AlP_3O_9, the aluminum metaphosphate glass, consist of PO_4 groups only incompletely interlinked with AlO_4 groups. With a ratio of O to (Al + P) equal to $9/(3+1) = 2.25$ they resemble constitutionally the commercial soda lime glasses $Na_2O \cdot CaO \cdot 6SiO_2$ with $O:Si = 14/6 = 2.3$. However, again this similar constitution is achieved in the absence of alkali. On this and other specific chemical differences are based the potential applications of multicomponent phosphate glasses such as the following: (1) enamels and "fluxes" of considerable resistance but low viscosity; (2) extremely resistant and dielectric glasses of normal viscosity; (3) alkali-vapor-resistant tubes; (4) hydrofluoric acid-resistant glasses; (5) ultraviolet-transmittant glasses (to about 200 mμ); (6) special colored glasses; and (7) as a specific case of (6), the neutral and highly transparent iron glasses of high heat absorption.

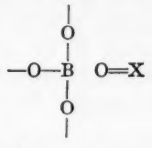
The usual green color caused by a combination of Fe^{2+} and Fe^{3+} in a silicate surrounding is not found in phosphates.

BORATE GLASSES

B_2O_3 forms an oxide glass quite different in constitution from SiO_2 or P_2O_5. It is a polymer consisting of BO_3 groups, which may be represented as triangles in a plane. This random network is more stable than any symmetrical arrangement conceivable. B_2O_3 and borate glasses are of high interest because of their low viscosity, high transparency to X-rays and ultraviolet light, and their relatively low dispersion. Their application is limited by the water solubility of B_2O_3 and many borates.

However, the addition of B_2O_3 to silicate glasses causes the solubility and expansivity to decrease to certain minima, and glasses of extreme usefulness are obtained. It may suffice to name porcelain enamels, vitrifiable colors on milk bottles, and laboratory glasses.

The constitutional cause of this agreeable contradiction is the fact that basic oxides, like Na_2O, permit boron to organize in BO_4 groups



resembling SiO_4 or AlO_4 groups.

Similar combinations exist between B_2O_3 and other oxides. The desire for unusual optical properties has focussed interest on borates free of silica or of low silica content. The recent combination of rare element oxides with each other and B_2O_3 has introduced lenses of very high refraction with relatively low dispersion. In the past, barium silicate glasses had been the best in this respect. The gradual substitution of B_2O_3 for Si_2O_4 in a barium crown produces a decrease in the relative dispersion $(n_F - n/n_D - 1)$ with rising refractive index n_D. In almost any other chemical substitution causing a rise in index, the relative dispersion increases also. The high B_2O_3 members of the series, however, are attacked by water acidulated with CO_2.

The corresponding lanthanum borates possess a very high index and a favorable ratio between index and relative dispersion. The acid resistance may be increased by permitting a certain silica content. Barium lanthanum borate glasses containing some silica and other oxides, such as zirconia, have a somewhat less favorable relative dispersion but a high refractive index and improved chemical resistance. Glasses of this type are the basis for more complex nonsilica glasses that have already proved their value in the photographic field. For general use in the field of optical instruments it is likely that glasses intermediate between the older silica glasses and the new nonsilica glasses will be valuable.

The desire for new glasses of exceptional properties will lead to a more thorough study of uncommon glass systems in the future. The fluoride, phosphate, and borate systems should be more thoroughly explored.

Instrumentation in Chemical Analysis[1]

A Technical Training Course

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THE number and variety of instruments used in chemical analysis have become so great that demands are being made on educational institutions for a course or courses of training to cover as many of these instruments as possible. Problems of finding sufficient classroom and laboratory space, of acquiring a minimum number of instruments for a general and fundamental study, and of selecting the proper students immediately confront the school that decides to initiate a course of instruction of this kind. It is obvious that the organization and teaching of such a course will tax the ingenuity of any instructor who attempts to do so, since it would be surprising if he should be thoroughly familiar with all the many instruments and the uses to which they may be applied in the field of chemical analysis.

Pedagogical methods may, in many cases, have to be altered from the orthodox, since it seems plausible that training should be based more directly on what the student is expected to encounter in the industrial laboratory than on his general knowledge of the subject. Thus an experiment should be designed in such a manner that the student must rely to a great extent on his own resources. The chemist in industry often meets a particular problem in which a new instrument may appear to be desirable. He must buy or make it himself. Having the instrument, he may find the problem necessitates alterations in the instrument or the way in which it is to be manipulated. The purpose of this course should be to give the student familiarity with the applications and limitations of the instruments studied, so that the correct choice may be made, and so that the needed alterations in the instrument or its application can be carried out.

In the industrial laboratory distinction should be made between the attendant, who is merely an automaton on a particular instrument, and one who has a knowledge of its fundamental action and thus is able to appreciate the results of his efforts. The analytical chemist need not be an instrument maker, but it would be extremely to his advantage to understand the construction as well as the functioning of instruments.

Ralph Müller in the May number of the *Analytical Edition* of *Industrial and Engineering Chemistry* points out that our analytical needs in industry are deviating considerably from present academic training. He points out that automatic analysis is becoming so popular that data collection and its treatment intrigues the analyst far more than the mechanics involved, and the latter should be left to a mechanical engineer or an electronics expert. It appears, however, that the separation he makes is too distinct, at least for research purposes, since a knowledge of the limits or versatility of a new instrument may easily lead to new fields for its use if they are realized by the manipulator.

It is obvious then that in conducting a course on the use of instruments in chemical analysis, many problems arise to confront the instructor. Such a course was started in the fall of 1944 at the Rochester Institute of Technology and was considerably improved last year. Although many difficulties have been overcome in making this an effective course, others still confront the instructor, and it is hoped that through our own experiences, and with information obtained from others conducting similar courses in schools throughout the country, it can be improved further.

The number desiring this course in Rochester is so great that a means of selecting students and instruments had to be found. A plan was worked out whereby candidates were selected from those employed regularly in local industries during the day. These students attend the course in the evening. A committee of chemists representing the many industries in Rochester serves in an advisory capacity to the Institute staff. This committee meets irregularly to discuss improvements, suggest added instruments, arrange for likely students, and give advice. An annual report is submitted by the instructor through the head of the Chemistry Department to the members of this committee on the achievements of the year.

The class of 1945–46 was conducted as follows: Eighteen students were chosen from the applicants, and six units were studied. A unit was considered a specific phase of instrumentation in chemical analysis. Three students were assigned to each unit. They were expected to cover their assignment in three sessions of two and one-half hours per session. Then they shifted to a second unit for three sessions, and so on until each group had covered all the units furnished.

The six units chosen were spectrophotometry, conductometry, potentiometry, amperometry, polarographic analysis, and spectrographic analysis. Spectrophotometry includes colorimetry and turbidimetry and ranges from 200 μ to 2500 μ. Amperometry and polarographic analysis were separated so that the former could include the dead-stop end-point experiments. The Bausch and Lomb large Littrow quartz

[1] Presented before the Division of Chemical Education at the 110th meeting of the American Chemical Society in Chicago, September 9–13, 1946.

spectrograph with auxiliary equipment gave enough range for a single unit.

Typewritten instructions were given each student at the outset to cover the entire course. The several experiments for each unit were on separate sheets so that they might be revised or other experiments substituted conveniently. An introduction of an explanatory nature was given for each experiment which included the objective of the experiment. Detail of instruction was varied. In some cases it was of the cook-book type to speed the student. Often the instruction was left incomplete and much was left to the ingenuity of the individual. For example, in the spectrophotometric determination of iron each step of the procedure was given and the experiment was completed rapidly. For analysis of nickel with the same instrument, the student was given a reference to instruction in the literature and told to proceed accordingly.

At the beginning of a new shift the instructor held a classroom session of not more than one hour for purposes of introduction and for questions concerning the previous shift. No attempt was made to cover theoretical considerations through classroom presentation. Such instruction was given to the individual group either by the instructor or his assistants. This arrangement is to be altered in the next class as will be outlined below.

Equipment for each unit was set up on a table of such size and shape as could conveniently be arranged about the room. Each table has a cabinet wherein all equipment not permanently attached and chemicals pertinent to that particular unit can be stored.

The nature of the apparatus used in the several units varied considerably and ranged from the most modern to some of the most simple, constructed of parts which could easily be obtained. This was considered good instructional technique, since the student could experience the distinction and could learn a great deal about the construction as well as the handling of instruments. For example, in place of an automatic recording polarograph, a simply wired apparatus mounted on a panel with an open back was constructed. The wires connecting galvanometer, voltmeter, rheostats, Ayrton shunt, etc., were fitted with patent clips for attachment. The student was given a diagram of the electrical connections and told to wire it himself; however, it is planned to have one automatic recording instrument in the course since the student should come to know that not all analytical procedures need be drudgery and also to show the advantages in some cases of securing permanent data independent of the analyst.

The course was conducted previously by an instructor and two assistants, one giving his entire time to the spectrograph unit. The course for the next class is to be extended considerably. The one criticism voiced most often was that the course was too superficial and that the student in general was still too little trained to carry on independent analytical procedures in the plant where he was employed. With this in mind the course this year will consist of two sessions a week for 25 weeks. One session will consist of lectures, demonstrations, visits to industrial plants, and attendance at pertinent scientific meetings. In general this session will be of one and one-half hours' duration. The second session of two and one-half hours will be conducted as before but with a minimum of fundamental instruction on the theory underlying instrumentation.

The rotational procedure in the laboratory will continue with six units and three students per unit, but with added sessions. The first session will be an organization meeting. It is expected an instructor and one assistant will be adequate for the laboratory. The classroom work necessarily cannot parallel work in the laboratory, for students are covering all phases of the course at any one time. This may be the most serious problem to contend with in this arrangement.

An attempt will be made to add at least one new unit each year. This year we propose to give chromatography as a new feature. The committee from industry referred to above recommended that in addition to the units now being taught, others, including density determination, viscosimetry, analysis by electrodeposition, refractometry, polarimetry, the microscope, and use of Geiger counters, be included eventually. Recent instruments which are very important also being considered are the electron microscope, the mass spectrograph, instruments for dielectric constant measurements, for electron diffraction, and for infrared spectroscopy.

An attempt was made this spring to secure information on how courses of this nature were being conducted by other educational institutions. A poll on instrumentation in general had been made by Mr. J. F. Sullivan, editor of *Instrumentation*, who kindly furnished us with a copy of his tabulation. This poll covered 88 schools. Thirty of these schools were selected as stating they were teaching courses in industrial instruments and the questionnaire was sent to them.

Questions were asked concerning when the course was initiated, whether it is specifically on instruments for chemical analysis, in what student year it is offered, whether it is offered in the regular curriculum or in evening school, the number and qualification of students, the length of the course, and, in particular, what instruments were being used. A question on the desirability of having the instructor from the industrial field rather than an educator familiar with the industrial field was also submitted.

Only about one-third of the schools returning the questionnaire stated they were conducting a course specifically for instrumental methods of chemical analysis. Others, however, stated the need for such a course. Where such courses were given, in general, only about ten students were being accommodated in a given class. The number of hours in classroom and laboratory was about the same as at the Rochester Institute of Technology. The type of instruments either recommended for such a course or actually being

used also did not differ greatly from those used or contemplated in this course.

In fact the only novel feature of the course at Rochester Institute of Technology seemed to be the rotational method of instruction in the laboratory whereby nearly double the number of students can be handled with the same teaching staff. As previously stated, this procedure in the laboratory is at variance with the lecture and demonstration procedure. This may not turn out to be a problem when put into actual practice.

One question submitted in the questionnaire concerned the treatment of data with respect to its interpretation. Not one answer was received to this question. Considerable stress is given to this phase of instrumentation in this course, and more time will be devoted to it in the more extended course.

From the returned questionnaires, it would appear that very few schools conduct a course specifically on instruments in chemical analysis; however, in some cases the query did not reach the proper individual in a given school and it is likely that other schools were overlooked. A follow-up of this venture would, it is believed, yield further worthwhile information, as it is certain that the demand for instruction on instruments in chemical analysis in educational institutions will greatly increase.

Conversion of a Bomb-Release Control to Activate a Solenoid Stirrer

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A GREAT deal of surplus army goods is being given to educational institutions for instructional purposes. The device described below is a bomb release control which can be easily modified for use as an intermittent energizer of a solenoid-type[1] stirring mechanism used in freezing-point determinations. To insure accurate freezing-point data, the time interval between each activation of the stirrer must be short enough for adequate agitation of the liquid.

The time interval in the two types of bomb-release controls described below may be varied between 0.08 of a second and one second. Within this range an operator can find an interval that will sufficiently agitate the liquid under observation.

The two types to be described are type AN-B3 which will be called Type I and type B-2A which will be called Type II.

Type I, the wiring diagram of which is shown in Figure 1, operates by charging and discharging condensers through resistances and relays. No tubes are employed. Type II (Figure 2) employs two 2050 thyratron tubes which regulate the current energizing the relays. The time is controlled by the charge and discharge rate of condensers through resistances in the usual manner.

The primary change necessary in each type is to prevent the control from automatically turning off after a certain number of impulses (number of bombs released). This is best accomplished in Type I in the following manner. Open the case and locate the cog wheel attached to the dial marked "bombs to be released" on the front panel. When this dial is at zero, a pin attached thereto opens the contacts of a jack. Unsolder the wires attached to the jack and resolder the wires together,

[1] BOOTH, H. S., AND D. R. MARTIN, *Ind. Eng. Chem., Anal. Ed.*, **17**, 528(1945); *Chem. Revs.*, **33**, 57 (1943).

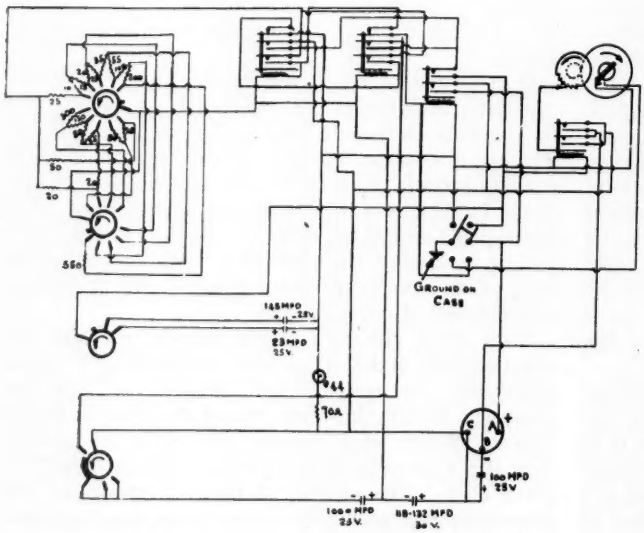


FIGURE 1. WIRING DIAGRAM OF TYPE I.

leaving the jack free. The control will now run continuously.

A similar result is accomplished in the Type II control in the following manner. Open the case and locate the cog wheel attached to the dial marked "bombs to be released" on the front panel. Locate the relay which operates the lever engaging this cog wheel. Unsolder one of the wires leading to the coil of the above relay. The control will now run continuously.

For use as an activating device for a solenoid or other similar use the bomb controls are connected according to diagrams in Figures 3 and 4. The connection indicated by a dotted line in Figure 3 is not absolutely necessary. Furthermore if a 24-v. d.-c. source is connected to Type I pin *A* and ground, any other device may be controlled by using pins *B* and *C* in series with the device and its power source. For Type II control a 24-v. d.-c. source must be connected as indicated by pins numbered *1*, *4*, and *5*. Any device may then be controlled by using pins *2* and *5* in series with the device and its power source. The 24-v. d.-c. source can be obtained by use of a lampbank on a 110-v. d.-c. line or with a 24-v. battery.

To operate Type I, a toggle switch marked S_1, Figure 3, must be put in the circuit and closed. The opening of this switch shuts off all power. Type II is operated by momentarily touching a tapping key shown in Figure 4. To shut off this type, the dial marked "bombs to be released" must be turned to zero. For both types the toggle switch on the panel should be left on "train."

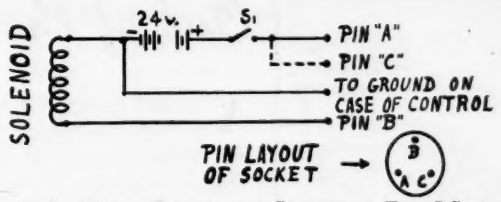


FIGURE 3. WIRING DIAGRAM FOR CONNECTING TYPE I CONTROL TO SOLENOID AND TO THE 24-VOLT D.-C. SOURCE

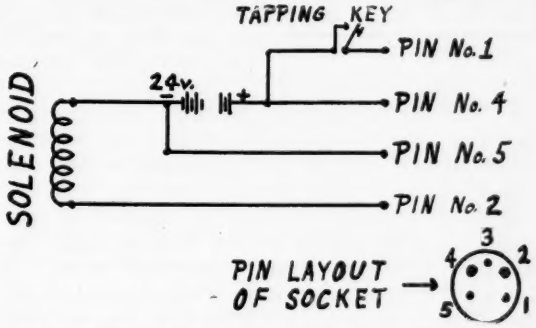


FIGURE 4. WIRING DIAGRAM FOR CONNECTING TYPE II CONTROL TO SOLENOID AND TO THE 24-VOLT D.-C. SOURCE.

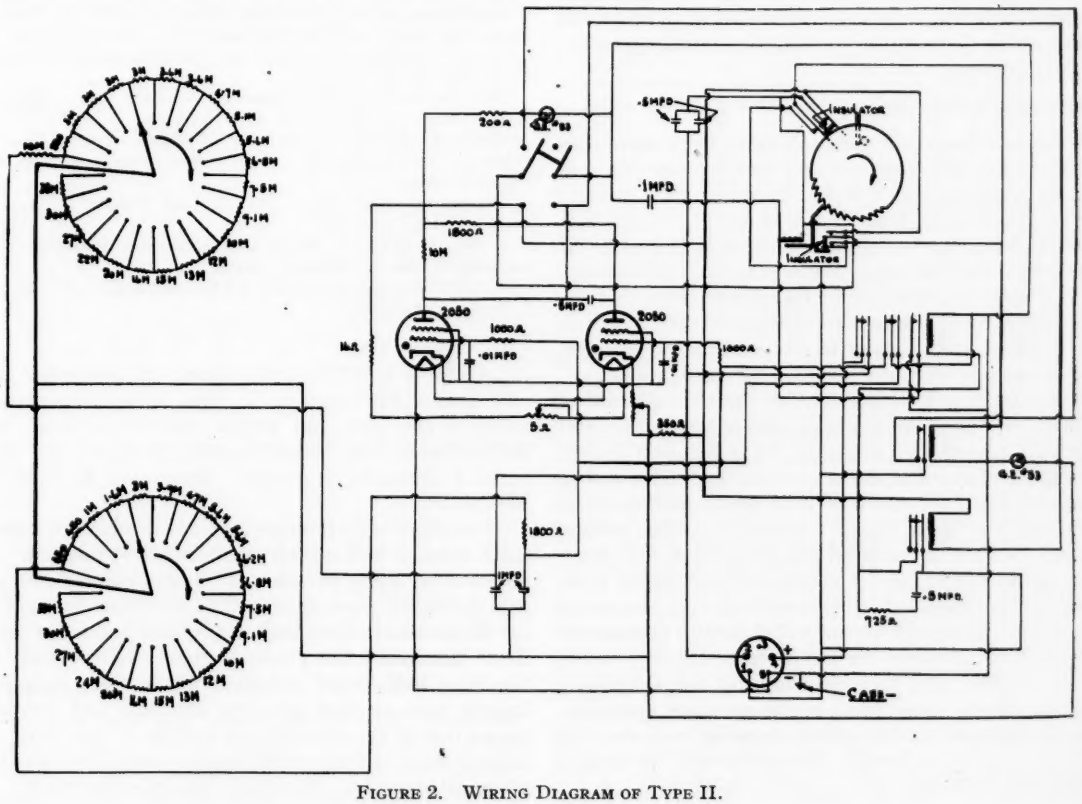


FIGURE 2. WIRING DIAGRAM OF TYPE II.

Out of the Editor's Basket

A CORRESPONDENT in India, Professor Arcot Wiswanathan of the Madras Christian College, sends us a description of two lecture demonstrations, one on gas diffusion and the other on osmosis. "Cartesian divers" are used as the means of indicating changes in pressure in each case. For the diffusion demonstration, two divers are placed in water in a tall glass cylinder, one being adjusted barely to sink and the other barely to rise. The cylinder is fitted with a stopper carrying a glass tube leading to a porous cylinder, as customarily used. Immersion of the porous cylinder in gas of high or low density will cause the divers, respectively, to sink and rise. The osmosis demonstration is a little more complicated, since the diver (which has been adjusted barely to rise in the solution) is placed in the osmotic solution in a glass tube of about 18 by 3.5 cm. This is closed at one end by a cellophane membrane and at the other by a stopper and a glass tube with a stopcock. Immersing the apparatus in water with the stopcock open, the level in the small tube is adjusted until the diver does not quite sink, then the stopcock is closed. Now when immersed in hypo- and hypertonic solutions, the diver will sink and rise.

Potassium Perchlorate

Potassium perchlorate, necessary in large quantities for success of the Navy jet and rocket programs, was usually imported before World War II. New producing units had to be built in this country, and the first West Coast plant was constructed in Los Angeles in 1941 by the Western Electrochemical Company. Today, after construction of many new plants, potassium and sodium chlorate are being produced at a rate one hundred times that of the original prewar installation to satisfy a world demand for this product.

The essential raw materials for the manufacture of chlorate and perchlorate chemicals are salt, water, and electrical energy. A saturated brine solution is prepared and adjusted to the proper acidity. This is then electrolyzed in a battery of cells under uniform temperature conditions and circulation. The sodium chlorate is then crystallized out of solution and separated from the liquor by means of a centrifugal filter. Some of this chemical is dried, milled, and packed for sale. The remainder is converted to the perchlorate by dissolving in water the wet chlorate salt as it comes from the filter, and then electrolyzing this solution in cells similar to those used in the previous operation. The sodium perchlorate solution coming from the cells is then converted to the potassium salt by adding potassium chloride; the product is then dried.

Employment Clearing House

With the opening of a New York regional employment clearing house for chemists and chemical engineers, the American Chemical Society has completed organization of a nation-wide employment service for the chemical profession with offices in seven key cities. Similar in purpose to the clearing house conducted at the Society's national meetings each spring and fall, the regional service will function throughout the year as a liaison agency, bringing together employers looking for chemists and chemical engineers, and Society members and student affiliates desiring such contacts.

The New York office, which will serve the Middle Atlantic region, has been set up in Room 93 in the Chemists' Club Building, 50 East 41st Street. The service has its headquarters in Washington at the American Chemical Society Building, 1155 16th Street, N.W., Washington, D. C., and also maintains offices in Atlanta, Boston, Chicago, Houston, and San Francisco.

Through the facilities provided by this chain of offices, an employer anywhere in the United States can obtain information about any chemist or chemical engineer in the country who is seeking employment by visiting the nearest regional clearing house. Records of all registrants are available in duplicate form at all seven offices, so that an employer examining the data at one office will be able to make a complete canvass of available manpower in his field.

By the same token, any of the Society's 48,000 members who are looking for a position has only to register with the service to place himself under consideration for any opening in the country which he is qualified to fill. Application blanks are available at all offices.

In addition to the New York office, of which Dr. Walter Baeza is in charge, and the Washington headquarters, under the direction of William B. Lodder, the following regional clearing houses are now in operation:

New England—Boston, Room 503, Boston University, 84 Exeter Street, Dr. Chester M. Alter in charge.

Southeast—Atlanta, Room 153, Chemistry Annex, Georgia School of Technology, Third and Fowler Streets, Dr. W. M. Spicer in charge.

Midwest—Chicago, Room 744, 35 East Wacker Drive, Dr. Marvin J. Hall in charge.

Southwest—Houston, Room 212, Chemistry Building, Rice Institute, Dr. W. O. Milligan in charge.

Pacific Coast—San Francisco, 585 Howard Street, Dr. Sterling L. Redman in charge.

Fluorine

To supply available information on properties and precautions for handling fluorine to purchasers and users of this rare and highly reactive element, the Pennsylvania Salt Manufacturing Company has prepared a preliminary manual, designated as *Pennsalt Manual F-1*.

Since this is a preliminary manual to meet an immediate need, it will not be given broad circulation. In addition to going to research laboratories selected for first deliveries, the manual will be distributed to a small list of industrial firms and universities and a few technical libraries. The manual gives instructions for handling half-pound cylinders of fluorine, including known data on first aid, the chemical and physical properties of the element, an outline of literature on organic fluorine compounds, and an introduction to the chemical literature of inorganic fluorine compounds.

Movies

Two new educational color and sound movies portray man's age-long struggle against weeds and insects, which yearly cost hundreds of millions of dollars and untold human suffering and annoyance.

The films, "Good-By Weeds" and "Doomsday for Pests," were made by Jerry Fairbanks, Inc., producers of "Popular Science," "Unusual Occupations," and other well-known shorts. Location scenes were shot in Hollywood, Cleveland, and New York.

"Good-By Weeds" opens with an early caveman, who has been able to provide himself with food, shelter, and clothing with his bare hands, but who is defeated in the end, not by the elements or wild beasts, but by the lowly weed. The color film then traces the struggle of the paleolithic man's successor against weeds through Biblical and early American Colonial times to the modern "push-button" era of living. The film unexpectedly reveals that modern man, in the atomic age, is using practically the same weapon against weeds that the cavemen, in the stone age, fought with.

After this review of war against wild plant life "Good-By Weeds" reviews the role that science is playing in making our day-to-day life a happier one. It goes on to point out that science has been concerned with weeds and recently developed a super-weapon against them. The weapon is dichlorophenoxyacetic acid, commonly called 2,4-D. This chemical compound makes it possible to destroy weeds merely by spraying them with water on lawns, golf courses, vacant lots, and other places ridden with weeds. Fortunately, 2,4-D is selective in its killing effect; it does not harm common lawn grass.

This weed-killing movie is particularly timely today, since campaigns are being conducted throughout the country to eliminate ragweed, poison ivy, thistle, marijuana, and other weeds, forecasting a happier future for hayfever sufferers and picnickers.

Live action and animation are combined in "Doomsday to Pests" to describe the effectiveness of DDT on insect pests. Novel treatment is given by showing the war against insects from the bug's viewpoint. The narrator is an insect, and, in a voice of doom, he tells the bug-world what they can expect from DDT. Live action then shows how DDT should be used. It explains its development and the terrific job it played during the war.

Applications for these movies will be handled through the Sherwin-Williams Company, its 350 branches, and its six affiliated companies—Acme White Lead and Color Works, W. W. Lawrence Company, the Lowe Brothers Company, John Lucas & Company, the Martin-Senour Company, and the Rogers Paint Products Company.

Plastics in the War

For those who like "now-it-can-be-told" stories we recommend an attractive 63-page booklet entitled "24 Case Histories," published by the General Electric Company, which tells how plastics solved war problems.

Office of Technical Services

Few people realize that the Office of Technical Services, which has been a part of the Department of Commerce for nearly a year, is greatly enlarging its activities and has five operation divisions:

Inventions and Engineering, to encourage invention and advise inventors on financial, marketing, and patent problems.

Technical Advisory Service, which continues its wartime function of answering industry's technical questions.

Bibliographic and Reference, to receive, abstract, and list declassified technical documents, to publish abstracts, and to provide indexing and reference service.

Technical Industrial Intelligence, which investigates science and industry in enemy areas. More than 500 investigators have already been sent to Germany, and a similar program is planned for Japan.

Industrial Research and Development, to finance worthy industrial research projects. This Division has recently issued a statement of its purposes, aims, and objectives, which we here reproduce:

Effective July 1, 1946, there has been established in the Office of Technical Services, Department of Commerce, an Industrial Research and Development Division (IRDD). The new Division is the custodian and administrator of the contract records, research reports, and files of the former Office of Production Research and Development, transferred by Executive Order of the President, January 3, 1946, to the Commerce Department from the Civilian Production Administration, formerly WPB.

The IRDD has been organized to initiate and conduct research and development on inventions, processes, and mechanisms, to promote and encourage technological productivity, and to create new products and be of general benefit to industry and small business enterprise.

For this purpose, the Congress has appropriated a fund with which to finance worthy projects. The fund will be administered by IRDD under a policy which provides that no project shall be eligible unless there is a reasonable probability that it will foster and promote the National welfare and that it will (1) advance the technological productivity of the Nation, (2) create new enterprise, (3) create new employment, and (4) be of wide public consequence and benefit. Projects may not be undertaken (1) if they are within the field of budget responsibility of another Government Agency, and (2) if they may be equally accomplished to the same ends by private enterprise.

IRDD will operate essentially in the field of applied research as differentiated from the basic research operations contemplated in the legislation creating a National Science Foundation, now before the Congress. It is proposed that IRDD will place contracts for worthy projects with the research laboratories of the Land Grant Colleges and other universities, as well as with the various industrial research institutions of the country. Due regards, of course, will be given to the availability of the specialized personnel, equipment, and facilities necessary to the economic prosecution of any project.

The patent responsibilities and contractor privileges inherent in IRDD contracts have been given a great deal of consideration. IRDD contracts will contain thoughtfully and carefully designed patent provisions to provide the contractors, the inventors, and the coworkers with the incentive to invent and present new ideas to the Government.

In general, it will be arranged for individual inventors of meritorious projects to accept employment with a university or other research laboratory under conditions which will permit them to receive the benefits of association with specialized expert personnel and the best research facilities. The Contractor-

Inventor will be permitted to retain the patent rights to any invention which is made as a result of the contract and with the use of Government funds.

It is planned that the Government contract in these circumstances will be placed directly with the research laboratory and that the contractor's employees and the inventor will work under such an agreement with the Contractor as will enable the laboratory to meet its contractual responsibility to the Government as to patents and other requirements.

The Contractor will, however, be required to grant to the Government the usual irrevocable, nonexclusive, royalty-free license to the Government to make, use, or sell the invention for Government purposes according to law, and also to give the Government a license on any other inventions made prior to the contract, upon which the contract invention may be dependent.

Should the Contractor wish to retain the invention rights, he is required to apply for the patents at his own expense. He shall also agree to license his new patent and any controlling patents to any qualified and responsible applicant under terms which are to be agreed upon by the contractor and the applicant. Terms shall be settled by arbitration if necessary.

IRDD will assist the Contractor to find a market for his invention and get it established industrially, for in the last analysis, this purpose is at the bottom of the Commerce Department policy.

Before undertaking any contract, the Government will make a thorough and careful evaluation to determine as far as possible the probability of its success. A Review Board will pass on the merits of each proposal. If the project has been well selected, benefits will accrue to the Contractor both from the licensing of his invention to others and from his own industrial application of it. Wide benefit should also accrue to industry from the licensing plan.

The contractor on a successful project will be required to return to the Government the sum of money that is properly chargeable to the part of the contract that was directly connected with the invention. Payments shall be made to the Government at the rate of one-half of industrial earnings and receipts from royalities until fully paid.

These are the highlights of the plan of operation as far as the Division has had opportunity to discuss them with the research laboratories and the inventors who are interested in owning their own patents. The Government will, of course, reserve its usual right to take out patents in the public interest and at Government expense if any of its Contractors do not wish to act in their own behalf.

Altogether, we believe that the inauguration of the IRDD is a constructive and promising addition to the national industrial economy. Our office is now prepared to consider any projects which are thought to conform with the acceptability requirements of the Division and shall be glad to discuss details on request.

ELY C. HUTCHINSON

Beryllium

Beryllium production processes, used by the Deutsche Gold and Silber Scheide Anstalt at Frankfort, Germany, are described in a report now on sale by the Office of Technical Services, Department of Commerce. Before operations were halted at the end of the war, the plant had achieved a maximum monthly production of 160 to 180 kilograms of pure beryllium.

The manufacturing process was divided into two parts—production of pure anhydrous beryllium chloride from the mineral beryl, and electrolytic production of metallic beryllium. In the first part of the process, beryl ore was ground in a dry ball mill, mixed with lime in a drum, and then fused in a rotary kiln. To separate the silica and lime from the other oxides present, the slag was disintegrated by a water quench and then dried. The dry clinker was treated with sulfuric dioxide to form sulfates of the soluble oxides.

Silica and calcium sulfate were removed by filtration. A hot ammonium sulfate solution was then added to the hot filtrate, and the alum in the mixture was crystallized out. Iron salts were removed from the solution by adjusting the acidity and by the addition of hydrogen peroxide and calcium carbonate. A sludge of ferric hydroxide and calcium sulfate was precipitated and removed by filtration.

The resulting solution was pumped into a circular-stirred vessel, and ammonia gas was passed into the solution until all the sulfate was neutralized and beryllium hydroxide was precipitated. The precipitated beryllium hydroxide was filtered, dried, briquetted with organic binder and charcoal, extruded in the form of rods, carbonized, and then chlorinated. Purification of the resulting crude beryllium chloride by distillation followed.

The electrolysis process was carried out in nickel crucibles with external auxiliary heating. The melt consisted of a mixture of equal parts of beryllium chloride and sodium chloride. The metal content of beryllium obtained from the electrolysis process generally ranged between 98 and 99 per cent, according to the Germans.

National Teacher Examinations

The American Council on Education has announced the eighth annual administration of its National Teacher Examinations. These tests will be administered in official examining centers throughout the United States on February 8 and 15 in 1947.

Arrangements are now being made for the establishment of examining centers in connection with school systems and collegiate institutions engaged in teacher education.

The National Teacher Examinations were made available by the American Council on Education as an aid to administrators in their efforts to improve the selection of teachers. The tests included in the battery are designed to provide objective measurement of certain of the abilities and knowledges of prospective teachers. They measure the intellectual, academic, and cultural backgrounds of prospective teachers, and are used in combination with records of experience, academic marks, ratings in various aspects of personality, etc., in the evaluation of an individual's qualifications for teaching.

Superintendents and Boards of Education in many localities require teaching applicants to present National Teacher Examination records. The examinations are also used in colleges as qualifying examinations and as guidance instruments to provide additional bases for student self-study of individual strengths and weaknesses in areas measured by tests.

The sponsors of the program emphasize the fact that misuse of the examination results as a sole basis for teacher selection is opposed. Records of experience, academic marks, ratings of various aspects of personality, etc., should be considered in addition to the National Teacher Examination record in the evaluation of a prospective teacher's competency.

Used in combination with additional information of the types named, it is believed that the National Teacher Examination results can contribute materially to the attainment of education's fundamental goal, the provision of the best possible instruction for young people in the schools.

Arrangements for cooperation in the Teacher Examination project may be made by writing to Dr. David G. Ryans, Associate Director, National Committee on Teacher Examinations, American Council on Education, 15 Amsterdam Avenue, New York 23, New York.

Impulse Scaler

A new scaling device for the counting of beta particles and gamma rays in radioactivity research has been put on the market by Instrument Development Laboratories, 817 E. 55th St., Chicago 15, Illinois. The new Model 161 Scaler is expected to find extensive use for tracer research work in metallurgy, medicine, food technology, chemistry, and related fields. It operates on impulses from a Geiger-Müller tube and actuates an external register once for each 64 impulses it receives.

Alcohol

Ethyl alcohol (basic ingredient for hangovers), now being made for industrial use from petroleum gases, is shown by chemical tests made by the Standard Oil Company of New Jersey, Louisiana Division, to have fewer impurities than the beverage alcohol, made by fermentation. Synthetic alcohol, which is made from waste petroleum ethylene gas, was recently shown by ultraviolet absorption, an extremely sensitive chemical test, to have negligible proportions of impurities.

A great demand for alcohol during the next few years is foreseen and it is predicted that demand will exceed the supply for several years to come. For economic reasons it seems likely that in the future all grain alcohol will be used for beverage purposes. Whiskey distillers during the war turned to the production of industrial alcohol, but now have resumed the production of liquors, the stocks of which have been largely depleted.

The civilian use of alcohol in solvents, plastics, film, and transparent wrapping paper may be expected to exceed prewar consumption because of the excessive demand for consumer goods during this period. Also new uses for alcohol may be expected.

Silicones for Vacuum Pumps

A bulletin issued by the Dow Corning Corporation (Midland, Michigan) describes DC 702 and DC 703, two silicone fluids for use in high-vacuum pumps. Their principal advantage is said to be their stability to decomposition when exposed to the air at operating temperatures.

X-Ray Spectrometer

A booklet issued by the North American Philips Company (100 East 42nd St., New York City) describes their "Geiger-Müller X-Ray Spectrometer." The spread of application of this technique makes it important to keep up with the development of instrumentation in the field.

Quantitative Radioactivity

Another instrument in the same general category as that above is the Autoscaler, an instrument to make possible quantitative measurements of radioactivity in connection with Geiger-Müller tubes. This is one of several developments from a new organization, Tracerlab (55 Oliver St., Boston, Massachusetts).

Alumina from Clay

A high-grade alumina, for the manufacture of aluminum, has been extracted from clays and low-quality bauxites by Chemical Construction Corporation at a plant which it designed and constructed for the U. S. Government at Salem, Oregon.

A great deal of interest has been displayed during the past ten years in the possibility of such extraction because high-grade deposits of bauxite in the United States were being depleted rapidly. This was especially true during the war when the demand for metal grade alumina was at extraordinarily high levels, making it imperative that a new method be found to utilize the aluminous clays and low-grade bauxites existing in large quantities in the United States. Without such a process the country would be dependent on foreign bauxities which, in an emergency, would be useless.

A reasonable quantity of high-grade alumina meeting metallurgical specifications in every respect has been produced in this plant after approximately eight months of test operations. Because of the war's end and the availability of an uninterrupted supply of imported bauxite, however, it has been decided to cease operation and place the equipment in stand-by condition. This new process of producing alumina, in the opinion of engineers, may prove valuable to countries having aluminous clay but no bauxite and which seek to establish their own aluminum industries.

The process uses ammonium bisulfate as the active reagent. There are nine principal steps in the process: disintegration and dehydration of the clay; fusion of commercial ammonium sulfate in an electric furnace for the production of ammonium bisulfate; digesting the dehydrated clay in a saturated solution of hot ammonium bisulfate; reduction of dissolved iron in the solution; crystallization of the ammonium alum from the saturated leach solution, separation of this crystalline salt from the mother liquid, and oxidation and precipitation of iron; resolution of the ammonium alum and precipitation of aluminum hydroxide, cooling, filtering, and washing of the precipitated aluminum hydroxide; and conversion to alumina by calcination.

Alumina is recovered from the alum crystals by treatment with ammonia and this, apparently, is the crux of the process. Former experimenters had considerable trouble preparing a filterable aluminum hydroxide, which is unusually gelatinous and difficult to process.

Division of Chemical Education

of the American Chemical Society
Chicago, Illinois, September 9–13, 1946

MINUTES OF THE EXECUTIVE COMMITTEE MEETING

Roosevelt Room—Morrison Hotel
Tuesday, September 10, 1946, 5:00 P.M.

Present: L. L. Quill, *Chairman*, John Cavelti, *Vice-Chairman*, John Timm, *Member at Large*, C. E. White, *Treasurer*, Paul H. Fall, *Secretary*, N. W. Rakestraw, H. N. Alyea. Also present were Norman Hill, O. M. Smith, and Arthur Schroeder.

1. Dr. Quill reported that some of the officers and members of the Division had participated in the selection of scientific apparatus for colleges and universities in the devastated countries of Europe. This was in response to an urgent request from the State Department. This required a great many hours and days of work, with frequent meetings in New York, Chicago and Washington.

2. Mr. Norman Hill, who has sponsored four symposia on technical library techniques, orally requested that a section, to be known as the Technical Literature Section of the Division of Chemical Education, be authorized by the Executive Committee of the Division.

After brief discussion, the Executive Committee felt that this request could not be acted upon until a complete write-up of the proposal was furnished and time allowed to study the proposal and its implications. Subsequently it would be necessary for the Executive Committee to confer with Mr. Alden Emery, Secretary of the American Chemical Society, concerning legal factors involved in the formation of such a section.

3. Dr. Arthur Schroeder of the U. S. Department of Commerce brought before the Executive Committee an announcement of the program of the Industrial Research and Development Division of the Office of Technical Service, which he felt would be of interest to members of our Division. He was invited to present this program to the members of the Division at its business meeting the following day.

4. The Executive Committee received a letter from Dr. O. M. Smith of Oklahoma A. & M. College, tendering his resignation as Chairman of the Committee on Examinations and Tests. This was accepted with regrets and also with expressions of genuine appreciation of the splendid work he has done as Chairman of that Committee for the past sixteen years.

5. On motion by C. E. White, seconded by H. N. Alyea, the Executive Committee voted unanimously that Dr. Theodore Ashford of the University of Chicago be made Chairman of the Committee on Examinations and Tests. Dr. Ashford has for several years served as a valuable member of Dr. Smith's Committee.

6. Dr. C. E. White made a motion, seconded by Dr. J. E. Cavelti, that Dr. O. M. Smith of Oklahoma A. & M. College be made Chairman of the Committee to Study Problems of Teachers of Chemistry in Colleges and Universities. Motion carried. This Committee was authorized by the Division at the Atlantic City Meeting in 1941, but because of the war and other complicating factors of less global significance, no appointment had been made until this meeting. Dr. Smith is to select the members of his Committee.

7. The Executive Committee, on motion by Cavelti, seconded by White, voted to appropriate $300.00 for the preliminary expense of the work of this Committee to Study Problems of Teachers of Chemistry in Colleges and Universities.

8. The Executive Committee elected the following men to be members of the Board of Publication of the JOURNAL OF CHEMICAL EDUCATION: Dr. L. E. Steiner, Oberlin College. Term to expire September, 1948. Dr. L. L. Quill, Michigan State College. Term to expire September, 1949.

9. On motion by Alyea, seconded by Cavelti, it was voted that Elbert C. Weaver's Committee on High School Chemistry be continued as constituted for the past year in accordance with his request. It was also voted that the original appropriation of $100.00 for the use of Weaver's Committee (voted by the Executive Committee at its meeting in Atlantic City in April, 1946) be continued for the year 1946–47, since he had not drawn on the Treasurer for any of this original appropriation.

10. The Committee approved the membership of the Committee on Examinations and Tests as submitted by the new Chairman, Dr. Theodore Ashford.

11. Dr. C. E. White, *Treasurer*, presented his annual report which was accepted, subject to recommendation of an auditing committee. The Treasurer's report is appended to this report of the Executive Committee.

12. The Executive Committee spent considerable time discussing plans for future programs of the Division, particularly for the Atlantic City Meeting next April 14–18, 1947.

13. The meeting adjourned at a late, but un-noted, hour.

PAUL H. FALL, *Secretary*

REPORT OF TREASURER, SEPTEMBER 1, 1946

Receipts:		
Balance September 1, 1945	$ 392.61	
Dues 185 members	185.00	
Dues for 1947–48, paid in advance	5.00	
Interest on savings account	1.35	
Interest on war bond	7.50	
From Chemical Education Publishing Company for JOURNAL OF CHEMICAL EDUCATION	4618.72	
Total receipts		$5210.18
Expenditures:		
Envelopes, University of Maryland Press	3.00	
Stamps, Postmaster, College Park, Md.	6.00	
Secretarial, Treasurer's Office, A. C. Engle	8.10	
Allotment to Secretary's Office, P. H. Fall	50.00	
A. C. S. News Service, Atlantic City abstracts	14.25	
Dinner for slide operator, Atlantic City	3.00	
Total expenditures		$ 84.35
Balance on hand		$5125.83
Assets are distributed as follows:		
Checking account, "Division of Chemical Education," Prince George's Bank and Trust Company, Hyattsville, Maryland		$ 413.28
Savings account, same as above		4712.55
3 U. S. Saving Bonds, $100 each, Nos. C825778g, C825779g, C825780g, "Division of Chemical Education," deposited in the safe deposit box of C. E. White at Prince George's Bank and Trust Company, Hyattsville, Maryland		300.00
Total		$5425.83

CHARLES E. WHITE, *Treasurer*

MINUTES OF THE BUSINESS MEETING

Roosevelt Room—Morrison Hotel
Wednesday, September 11, 11:30 A.M.

1. The Secretary reported the action of the Executive Committee held the day before (see report immediately preceding).

By unanimous vote the action of the Executive Committee was approved by the members of the Division present at this Business Meeting.

2. Dr. C. E. White, Treasurer of the Division, gave his report and Dr. Frederick Dutton of Baldwin-Wallace College reported that he had audited the Treasurer's books and found them to be correct and in good order.

The report was accepted.

Treasurer's report is appended to the report of the Executive Committee.

3. Dr. L. L. Quill, Chairman of the Division, gave a brief explanation of the income from the Chemical Education Publishing Company and the increased appropriation made by the Board of Publication of the JOURNAL OF CHEMICAL EDUCATION for editorial expenses and an increase in compensation for editorial services.

4. Dr. J. Sam Guy, of Emory University, reported his experience as a member of a University Committee dealing with surplus war property. He stated that educational institutions have fifth place in priority.

5. Dr. L. E. Steiner reported for the Nominating Committee:
L. E. Steiner, *Chairman*, Oberlin College, Oberlin, Ohio
Bruce Weidner, Middlebury College, Middlebury, Vermont
Norris Rakestraw, Scripps Institution, La Jolla, California

This Committee presented the following slate:
Chairman: J. C. Bailar, University of Illinois, Urbana, Illinois
Vice-Chairman: E. L. Haenisch, Villanova College, Villanova, Pennsylvania
Secretary: Paul H. Fall, Hiram College, Hiram, Ohio
Treasurer: C. E. White, University of Maryland, College Park, Maryland
Member at large: F. D. Martin, Purdue University, Lafayette, Indiana

Motion made, seconded, and passed that the report be accepted. The Chairman then called for any nominations from the floor but none was made.

Motion made, seconded, and carried that nominations be closed and that the Secretary cast a unanimous ballot for election of the officers submitted by the Nominating Committee. This was done and the individuals named above were declared elected.

6. Dr. Babor of New York University asked about the possibility of a symposium on the Content of the Freshman Course. Dr. Quill then reported on the discussions of the Executive Committee relative to future meetings, stating that one symposium planned for the Atlantic City meeting in April is a symposium on Revamping the Curriculum in Chemistry and in Chemical Engineering.

Dr. Quill also reported the deadline for material for the Atlantic City meeting.

The meeting adjourned at 12:05 P.M.

PAUL H. FALL, *Secretary*

OFFICERS AND COMMITTEES

EXECUTIVE COMMITTEE 1946–47

J. C. Bailar, *Chairman*, University of Illinois, Urbana, Illinois. Term expires September, 1947.
E. L. Haenisch, *Vice Chairman*, Villanova College, Villanova, Pennsylvania. Term expires September, 1947.
C. E. White, *Treasurer*, University of Maryland, College Park, Maryland. Term expires September, 1949.
P. H. Fall, *Secretary*, Hiram College, Hiram, Ohio. Term expires September, 1949.
F. D. Martin, Purdue University, Lafayette, Indiana. Term expires September, 1947.
L. L. Quill, *Immediate Past Chairman*, Michigan State College, East Lansing, Michigan.

COMMITTEE ON HIGH-SCHOOL CHEMISTRY

(Authorized September, 1939)

In September, 1946, by request of the Chairman, the membership was continued as constituted for the past two years.

Elbert C. Weaver, *Chairman*, Phillips Academy, Andover, Massachusetts. Elected Chairman September, 1943. No definite limitation on term. Subject to recall or reappointment by Executive Committee of the Division. By action of Executive Committee, approved by the Division, $100.00 was appropriated for expenses for this Committee. None of these funds was expended up to September, 1946, and by vote the amount appropriated was continued for year 1946–47.

T. A. Ashford, University of Chicago, Chicago, Illinois; T. D. Kelsey, Grover Cleveland High School, St. Louis, Missouri. Terms expire September, 1947.

A. D. Lincoln, Technical High School, Springfield, Massachusetts; Robert L. Ebel, Edison Institute, Dearborn, Michigan; Harold W. Baker, James Ford Rhodes High School, Cleveland, Ohio. Terms expire September, 1948.

T. A. Nelson, Decatur High School, Decatur, Illinois; R. D. Reed, State Teachers College, Montclair, New Jersey; Ernestine M. J. Long, Normandie High School, St. Louis, Missouri. Terms expire September, 1949.

COMMITTEE ON EXAMINATIONS AND TESTS

(Authorized about 1930)

T. A. Ashford, *Chairman*, University of Chicago, appointed by Executive Committee in September, 1946. No definite limitation on term. Subject to recall or reappointment by Executive Committee of the Division.

E. F. Degering, Purdue University, Lafayette, Indiana; Dale Dreisbach, University of Akron, Akron, Ohio; Gordon Pritham, University of Scranton, Scranton, Pennsylvania. Terms expire September, 1947.

Alex Calandra, Brooklyn College, Brooklyn, New York; Ralph Van Peursem, Rochester Athenaeum and Mechanics Institute, Rochester, New York; Frank Martin, Purdue University, Lafayette, Indiana. Terms expire September, 1948.

Conrad Ronnenberg, Denison University, Granville, Ohio; E. L. Haenisch, Villanova College, Villanova, Pennsylvania. Otis Dermer, Oklahoma A. & M. College, Stillwater, Oklahoma. Terms expire September, 1949.

COMMITTEE TO STUDY PROBLEMS OF TEACHERS OF CHEMISTRY IN COLLEGES AND UNIVERSITIES

(Authorized at Atlantic City Meeting in September, 1941)

Because of the war and other complicating factors of less global significance, no appointment was made to this Committee until September, 1946, at the Chicago meeting when Dr. O. M. Smith of Oklahoma A. & M. College was elected, by the Executive Committee of the Division, Chairman of this Committee with power to choose its members and with an appropriation of $300.00 for the preliminary expense of the work of this Committee.

COMMITTEE TO STUDY PROBLEMS OF TECHNICAL AND NONCOLLEGIATE CHEMISTRY

(Authorized by the Division at the Atlantic City meeting in 1941)

As yet no members have been named during the administrations of: R. D. Reed, retiring 1941; F. N. Brown, retiring 1942; A. J. Currier, retiring 1943; L. L. Quill, retiring 1944.

COMMITTEE ON HISTORY OF CHEMISTRY IN AMERICA

(Committee appointed at Board of Publication Meeting, September, 1941)

Norris W. Rakestraw, *Chairman*, Scripps Institution, La Jolla, California.

At the Memphis Meeting in April, 1942, a Committee was appointed to make a study and formulate a report on how the content of chemistry in the liberal arts colleges can be adapted to aid in problems confronting the nation in war time. Their findings were to be reported to the U. S. Office of Education. The Committee consisted of:

John E. Cavelti, *Chairman*, Allegheny College, Meadville, Pennsylvania; Sidney J. French, Colgate University, Hamilton, New York; Roy I. Grady, College of Wooster, Wooster, Ohio; Andrew J. Scarlett, Jr., Dartmouth College, Hanover, New Hampshire.

(Continued on page 623)

Report of the
NEW ENGLAND ASSOCIATION of CHEMISTRY TEACHERS

The Application of Colloid Chemistry to the Manufacture of Inflated Articles[1]

LANGLEY W. ISOM

Dewey and Almy Chemical Company, Cambridge, Massachusetts

NATURAL rubber latex—the juice of the rubber tree—consists of colloidal particles, each apparently made up of a core of hydrophobic rubber hydrocarbon surrounded by a sack of hydrophilic protein material which acts as a protective colloid or protective coating and prevents the particles from coalescing.

Chemists had not worked with rubber latex very long before they discovered that the particles carry negative electrostatic charges and that if a current is passed through the latex, a deposit of rubber can be obtained on the anode.

This suggested a process for the manufacture of rubber or rubber coated articles similar to that used in the electroplating of metals—simply have an anode the shape of the desired article, immerse it and its corresponding cathode in latex, turn on the current and, when the desired thickness of rubber is obtained on the anode, strip off the finished article.

Such a process might have made considerable progress had not an even simpler way of accomplishing the same thing been discovered. Because many polyvalent salts which produce strong cations will neutralize the charges on the latex particles and cause them to coalesce, why not coat a form with such polyvalent salt, dip it in the latex, and let nature do the rest? The idea was tried, it worked, and the latex dipping business was born.

Dipping of rubber aticles was done on a commercial scale well before the process had been worked out for latex—so-called solvent dipping. In this process the rubber is first milled and dissolved in a solvent, such as naphtha, to produce a thin rubber cement. A former of the desired shape is then dipped into the cement and almost immediately withdrawn. A thin film (about 0.002 inch thick) adheres to the former. It is dried and then dipped again and picks up another film of the same thickness. This means dip after dip before reasonable thickness for most articles is obtained. If it is attempted to get heavier films by increasing the solids content of the rubber solution, the solution becomes so viscous that is is impossible to do anything with it. With natural rubber it is not practical to dip in a solvent solution having a solids content of much over 10 per cent. Latex for dipping, on the other hand, can be prepared so it is perfectly fluid with a solids content of 50 to 60 per cent.

In preparing a solution of rubber, it is necessary to break down the rubber considerably on a mill before the solvent is added. This destroys the quality of the finished product. Latex dispersions, on the other hand, are not broken down at all and, consequently, produce rubber articles of very high quality.

Finally, solvents are volatile, expensive, and toxic. Water, which replaces the solvent in latex dipping, has none of these disadvantages.

These three advantages—greater film thickness per dip, better quality, and freedom from the objectionable properties of solvents—have caused latex dipping to replace solvent dipping except in a few special instances.

If articles with heavy walls are required, more than one dip can be made in latex to build up almost any thickness of film. One of the common ways of doing this is first to dip the former into latex compound and then into an acetone solution of a polyvalent salt, such as calcium nitrate. The calcium nitrate not only coagulates this latex film but leaves excess cations on the surface which can be used to coagulate another layer of latex into which the former is then dipped as soon as the acetone has evaporated.

This process can go on and on with alternate dips into latex and calcium nitrate. Each layer has much greater thickness than can be obtained by dipping in a rubber solution and, in subsequent curing, the layers are welded together so that lamination is only a minor problem.

An interesting fact about latex dipping is that the gel which is formed has just about the same solids content as does the compound from which it is deposited. This is hard to believe because the gel has many of the properties of solid rubber, whereas the compound is a liquid. What has taken place is not dehydration but an inversion of phase. Before coagulation the water is the continuous phase; after coagulation both phases become continuous.

[1] Presented before the Eighth Summer Conference of the N.E.A.C.T. at Middlebury College, August 22, 1946.

The dipping processes were, of course, originally worked out for natural rubber latex. When the synthetics came along, the process got a break in the fact that most of the synthetic rubbers are prepared by first making their latices and then coagulating them with acid to gain the solid material. This is so, for instance, in the case of the various types of Buna rubbers and in the case of neoprene. Neoprene has proved itself to be the synthetic best adaptable for dipping, and we are now using it exclusively. We formerly did some dipping with a latex of the Buna S type, but the quality of the resulting products was not outstanding. With neoprene we have been able to do everything which we formerly did with natural rubber latex, and the results have in all instances been as good and in some instances better.

The process which I have described may sound very simple and, in principle, it is. In practice, however, it takes as good a rubber chemist as there is in the business to work out the details and make the process practical on a commercial scale. All compounding agents, dyes, accelerators, and antioxidants must, of course, be thoroughly compounded with and dispersed in the latex before dipping is commenced. Some of these materials tend to destroy the colloidal stability of the latex and getting them into it and still having a mix which can be used for days or weeks is quite a problem. It is easy to say, "Coat the form with a polyvalent salt," but actually doing it can be a mighty tricky business.

Air is the latex chemist's greatest enemy and he gets gray hair devising methods for keeping air bubbles out of his compounds and out of the films of his products. Formers must be designed so that there are no pockets on their bottoms which entrap air, and great care must be exercised in immersing them in the latex.

After dipping we can do two things with the gel which we have formed. If we want to make an article just the size of the former we dipped, we can put the former in a drier with the gel still on it, leave it there until dry, then strip it from the former and vulcanize it. But if we want to make a balloon ten or twelve inches in diameter, we do not need such a former. A much smaller one is plenty big enough. First we strip the gel. Then without drying at all we put it on the nozzle of a blower and blow it up. Then we put a cork in its neck so it will not deflate. After it is dried, it is deflated and vulcanized.

I never cease to be astonished at the fact that a rubber gel which has just been formed and is still wet will inflate so well. What takes place during this inflation is not elastic stretching of the rubber; it is plastic flow—one rubber micelle actually flowing over the other with a consequent thinning of the walls. In contrast with the elastic stretching of rubber, this plastic flow is nonreversible.

So much for the process. You may now like to hear about some of the rather interesting products we make with it. At the present time we are using it to make football and basketball bladders, playballs, and balloons—both meteorological and toy.

We got into the meteorological-balloon business in 1935 when the Blue Hill Observatory of Harvard University asked us to develop a new type of balloon to carry the radiosonde on which they were then working. We were phenomenally successful in the development of this balloon, and it made possible the whole science of air mass meteorology.

It was not long before we were making not only this balloon but all balloons which meteorologists use. It may be interesting to you to know just how meteorologists obtain their data by means of balloons.

I am going to start with the smallest balloon which we make, the J-10 ceiling balloon, which is used to determine the base of clouds. It weighs 10 grams and is released at a diameter of about 18 inches. CAA regulations prohibit a plane's being cleared for an airport unless the prediction of ceiling at that airport is greater than 500 feet. This ceiling is usually determined with ceiling balloons and, consequently, these balloons are extremely important to all types of aviation. A ceiling balloon is inflated on a balance with hydrogen or helium until it has a lifting force (known from previous data) to give it a certain rate of rise. This rate of rise is entirely independent of horizontal drift caused by winds. The observer simply clocks the time between release of the balloon and its disappearance into the cloud base. This time indicates the height of the cloud base.

The next largest size is the J-30 pilot balloon which is released at a diameter of 27 inches and bursts at 40 inches about 35,000 feet up. This balloon is used to determine wind direction and velocity at various elevations in the atmosphere. It is perfectly possible to have a northeast wind at the surface and a southwest wind at 10,000 feet and, of course, these facts are of tremendous importance in weather forecasting.

Like the ceiling balloon, the pilot balloon is inflated to a certain free lift and released. However, in this case more than altitude is being determined; therefore the balloon is followed with a special form of transit known as a theodolite. A theodolite is a transit which, in addition to its azimuth scale for measuring horizontal angles, has a carefully calibrated vertical scale for measuring elevation angles.

The balloon rises at a constant rate, and from this rate the distance of the balloon from the ground can be determined at any instant. This gives one side of the right triangle formed by the observer, the balloon, and the point on the earth directly under the balloon. The theodolite's elevation scale measures another angle and thus makes it possible to solve the triangle and locate the balloon except for its compass bearing from the observer. This is read from the azimuth scale of the theodolite, and the exact position of the balloon can then be determined from tables which have been prepared beforehand. Readings of elevation and azimuth are taken every minute, timed by a buzzer, and are plotted on polar coordinate paper.

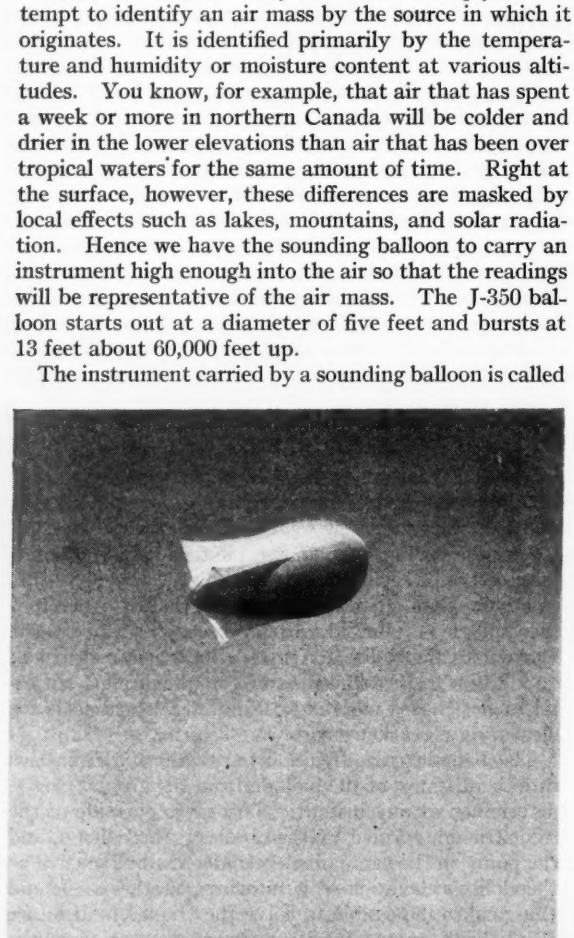
Like ceiling balloons, pilot balloons are made in various colors for maximum visibility. For night ob-

servation a small lantern containing a candle or a flashlight battery and bulb is suspended from the balloon.

The J-100 balloon, which is released at about 40 inches and bursts at six and one-half feet 50,000 feet up, is used exactly as is the J-30 but gets up faster and higher. It has its uses where observations to a certain height must be made quickly. It is also particularly useful on shipboard because, due to its faster rate of rise, it gets well up before it can be sucked into the vessel's air slip-stream.

The next standard balloon is the J-350 sounding balloon. To appreciate the work of this balloon, you must have some idea of the problem involved. For the last eight to ten years, meteorologists have been using what is called "air-mass" analysis. This is simply an attempt to identify an air mass by the source in which it originates. It is identified primarily by the temperature and humidity or moisture content at various altitudes. You know, for example, that air that has spent a week or more in northern Canada will be colder and drier in the lower elevations than air that has been over tropical waters for the same amount of time. Right at the surface, however, these differences are masked by local effects such as lakes, mountains, and solar radiation. Hence we have the sounding balloon to carry an instrument high enough into the air so that the readings will be representative of the air mass. The J-350 balloon starts out at a diameter of five feet and bursts at 13 feet about 60,000 feet up.

The instrument carried by a sounding balloon is called a radiosonde. A small parachute lowers the radiosonde when the balloon bursts. Inside the box are instruments for measuring pressure (which indicates height), humidity, and temperature. The box also contains a small radio transmitter. The instrument transmits by radio a continuous record of pressure, humidity, and temperature, the signals being picked up and automatically recorded by the ground station. All of these balloons which we are discussing are freely expanding—that is, they get bigger and bigger as they go up where the atmospheric pressure is lower and lower until finally they get so big they burst. The radiosonde falling from 60,000 feet could do considerable damage; hence the parachute. Even with the use of the parachute, the instruments are usually damaged when they fall, but a few of them are found and repaired.



Kytoon IN FLIGHT

This brings us to the biggest balloon which we now produce, the J-800. This balloon bursts at about 22 feet and will lift a five-pound instrument to 80,000 feet at about 1000 feet per minute. Carrying less weight, J-800 balloons have gone to over 100,000 feet.

It is important from many angles. Until about 15 years ago, meteorologists were satisfied with surface observations. Then they got data from 60,000 feet and found it useful. They now, quite naturally, think that data from 100,000 feet might be still more useful.

There is, however, a more immediate need for the J-800 balloon. A new instrument was developed during the war called "raywind." The raywind instrument incorporates a radiosonde and also those elements which make it possible to keep it in sight with directional radar measuring azimuth and elevation exactly as does a theodolite. This fulfills the meteorologist's dream of being able to get wind direction and velocity aloft when clouds prohibit the use of pilot balloons. There is, however, a snag in the raywind apparatus. A straight line drawn from the instrument to the ground station must not hit the earth at any intermediate point or the radar can't see the instrument. The earth being round, the balloon has to rise very rapidly so that the high winds aloft won't blow it below the horizon before the desired altitude is reached. This means faster rising balloons, of which the J-800 is the first. We expect the J-800 balloon to become standard with the weather services very quickly, but we already have an experimental order from the Army for a still larger balloon for special studies.

Our meteorological balloons are all designed for free flight and are not very satisfactory when flown captive. During the early part of the war, we were asked by the Signal Corps to develop a really good small captive balloon. We naturally took the job and after a hard battle developed a new balloon which we called the N-4.

The N-4 balloon looks like a J-350 but is compounded entirely differently and for different purposes. The J-350 has to go to 60,000 feet but must last only an hour and a half; the N-4 has to go to only 300 feet but must last for four days.

The N-4 balloon was designed to carry the antenna of the Gibson Girl—the portable radio transmitter

which was carried in planes to be used in a rubber life raft in case the plane had to be ditched. The Gibson Girl is so called because of its hourglass shape. With its balloon-borne antenna, its SOS can be heard for 1500 miles; without the antenna, the range would be insignificant. There are records of the rescue of well over 100 men through the use of the Gibson Girl.

The Gibson Girl also carries a kite. This is because any spherical balloon is unsatisfactory in strong winds. However, it is extremely hard to launch a kite from a life raft and a new single unit was definitely called for. We found the answer in our kite-balloon which we have called the *Kytoon*,[2] but unfortunately, we got it worked out just about in time for V-J Day.

The *Kytoon* consists of a streamlined casing made from lunes of very light Nylon fabric into which has been inserted a bladder made from Neoprene just like our other balloons. As its name implies, the *Kytoon* combines the good features of both kites and balloons. In a calm or very light wind it remains overhead by virtue of the static lifting force of the hydrogen or helium with which it is inflated, but given winds of 4 to 6 m. p. h., the static lift of the gas is completely masked by the *Kytoon's* kiting effect; it goes up, not down, as the wind increases.

Even in peacetime, we expect a good many *Kytoons* to be used with Gibson Girls which are now standard equipment on all transoceanic planes—military, naval, and civil. In addition to this use, there are many other possibilities for the *Kytoon* in sea rescue. All kinds of very high-frequency radio transmitters and radar responders are being developed by Army and Navy laboratories. These devices depend for their operation on being in line of sight of the receiving station. Consequently, a "sky hook" like the *Kytoon* becomes essential.

All of these military uses have been slow in coming because of constant changes in Army and Navy personnel during the last year. In the meantime, however, we have uncovered many other uses for the *Kytoon*. One of the most interesting is to carry a surveyor's target which can be seen through a transit over trees and hills. The target, supported by the *Kytoon*, is brought into position directly over a spot on the ground by pulling it with three guy lines until it is centered on the cross hairs of a vertical collimator. At that moment the operator yells "Mark" into the radio telephone.

[2] Registered trade mark.

Kytoon BEING INFLATED IN A LIFE RAFT

Two or more transit men, as far as ten miles away, hear him and clamp their transits onto the target, thus giving the bearings of the spot on the ground despite intervening obstacles.

We are also introducing the *Kytoon* as an aerial mark to fly above the forest canopy to mark areas to be sprayed with insecticides from the air. It has been proposed as a "sky hook" to carry aloft a small aerial camera. The Rockefeller Foundation is using the *Kytoon* to carry aloft aerial mosquito traps to study the migration of the yellow fever-bearing mosquito in the Brazilian jungle.

This tells you about our products except for the rather amusing story of how we arrived, much to our surprise, in the toy balloon business. We had always sold the seconds of our meteorological balloons to the street peddlers except during the war when Army Ordnance took all we had for target balloons. On V-J Day the peddlers were standing in line to get their seconds. They cleaned us out the first day. We were all geared up for big production, and the Army and Navy had terminated most of their contracts for meteorological balloons. Therefore, why not make first grade balloons for the peddlers until we could reconvert? We never expected the business to last but it did. The chain stores got hold of our J-30 balloons and we had to increase our facilities time and again until today we are making about five times as many J-30 balloons for the toy trade as we ever made for the Government.

DIVISION OF CHEMICAL EDUCATION (*Continued from page 619*)

COOPERATIVE COMMITTEE ON SCIENCE TEACHING

L. L. Quill was, in 1944, designated as the Representative of the Division of Chemical Education. No expiration date of term was designated.

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RECENT BOOKS

INTRODUCTION TO ATOMIC PHYSICS. *Henry Semat*, Ph.D., College of the City of New York. Second Edition. Rinehart and Company, Inc., New York, 1946. xi + 412 pp. 169 figs. 23 tables. 15 × 23 cm. $4.50.

This is one of the best books on this topic that has come to the reviewer's attention. As the title implies, the work is not intended for the expert in either nucleonics or spectral energy levels, but it contains a splendid review of the fundmantal knowledge in both these fields.

The subject matter is divided into three parts, of which Part I, entitled, "Foundations of Atomic Physics," deals with the following topics: elements of electricity and magnetism; elementary charged particles; electromagnetic radiation, waves, and particles. Part II, entitled, "The Extranuclear Structure of the Atom," consists of a chapter on the Bohr theory of the hydrogen atom, and another on atomic spectra and electron configurations of atoms. Part III is devoted to the discussion of the nucleus and consists of three chapters, the first on natural radioactivity, the second on disintegration of nuclei, and the third on nuclear energy.

In this last chapter the author has summarized the most recent information on fission, as set forth in the "vivid and dramatic report" by H. D. Smyth.

Finally, there is given, in nine appendices, a great deal of important data and derivations of some of the mathematical relations used in the previous chapters.

The exposition is extremely lucid and is aided materially by clear illustrations and diagrams. Furthermore, the publishers have done an excellent job in the printing and general make-up of the book. The reviewer can recommend it highly to chemists who wish to learn about the fundamentals of atomic physics without becoming too involved in the more highly theoretical questions, such as the nature of the forces between the particles in the nucleus itself.

SAUL DUSHMAN

GENERAL ELECTRIC COMPANY
SCHENECTADY, NEW YORK

MODERN CHEMISTRY. *A. J. Berry*, Fellow of Downing College, University Lecturer in Chemistry, Cambridge. Cambridge University Press, Cambridge, 1946. x + 240 pp. 14 × 21 cm. $2.50.

This book is devoted to the history of chemistry, as its subtitle, "Some Sketches of Its Historical Development," indicates. As to its general description, your reviewer is going to swallow his pride and quote *in toto* the "blurb" to be found on its jacket, for it is a vestpocket summary much superior to anything he could do himself.

"Teachers of chemistry in general realize the importance to students of a proper historical perspective, especially in those branches of chemistry where progress has been most rapid in the last 50 years. Existing histories, covering chemistry as a whole from the earliest beginnings, necessarily deal in too brief a fashion with the later work. Mr. Berry's plan is different. He makes no attempt to deal with the history of the whole of chemistry, nor does he make chronology the plan of his book; instead he devotes separate chapters to the history of separate subjects—electrochemistry; stereochemistry; radio-activity; elements; isotopes; experiments on gases; solutions; chemical change. By this method he is able to make the stories he has to tell of great theorists and experimenters in the recent past continuous and relevant to the present situation in each branch. The book is a contribution in support of the notion that a right attitude to any subject should include a knowledge of the order in which men have perceived and tackled the problems it presented."

The book lives up excellently to this advance description, and I recommend it without qualification to mature students. It is compact, well printed, and relatively cheap; the paper is not up to prewar Cambridge Press standards but is acceptable. It should have a long life on the shelves of those whose preoccupation with the current scene has not dulled their interest in how it was attained.

No one could write an interpretation of recent history without stirring up enough material for a dozen major controversies, but I do not consider this the proper place to open the debate. If the reader wishes to take issue with the author's choice of material, treatment, or placing of emphasis, so much to the good. He will be at least thinking, which is an improvement over cramming. Mr. Berry addresses those already endowed with "a moderate stock of chemical knowledge," but "moderate" is a word of various interpretations, and the chances of the reader's being so well endowed that he anticipates the writer, or fails to enjoy him, are small indeed. Most undergraduates will find it heavy going. I believe it would be hopeless as a textbook, and it is plainly not intended to be one. However, its nine chapters constitute nine essays of distinction, which will reward the careful reader. Not the least of their attractions is the smooth-flowing prose characteristic of the English school, pleasant to read and so difficult to duplicate. Chemistry makes claims to being a cultural subject, but it is only the occasional book, such as this one, which makes a convincing demonstration.

DOUGLAS S. CALDER

FOREST PRODUCTS CHEMICAL COMPANY
MEMPHIS, TENNESSEE

GAS IN LIGHT ALLOYS. *L. W. Eastwood*, Battelle Memorial Institute. John Wiley and Sons, Inc., New York, 1946. x + 99 pp. 34 figs. 2 tables. 14 × 21.5 cm. $2.50.

The presence of gases dissolved in metals or produced during casting by interaction of the metal with moisture in the mold has been a troublesome problem in the development of light metals and alloys. This book describes the way in which the gas contamination occurs, the methods of measuring gas contents and identifying the gas, the type of defects caused by gases and their effects on the properties of metal, and the techniques by which difficulties due to gases can be avoided. Gas contamination in aluminum receives fullest attention. The fact that the subject can be covered in a thorough manner in so few pages is cited as proof that "understanding is now rather complete." There are 59 references to the literature. The book will serve as a handbook for those who are working with light metals and alloys and should be in every metallurgical and chemical library.

LAWRENCE S. FOSTER

WATERTOWN ARSENAL
WATERTOWN, MASSACHUSETTS

QUALITATIVE INORGANIC MICROANALYSIS. *Ronald Belcher*, Rotherham Technical College, and *Cecil L. Wilson*, The Sir John Cass Technical Institute. Longmans, Green, and Company, New York, 1946. viii + 68 pp. 28 figs. 12 × 18 cm. $0.80.

A student laboratory manual, and a useful source of information on manipulative techniques for anyone having occasion to use micro methods. Pedagogically, it is based upon the firm conviction that micro methods of qualitative analysis are within the powers of average students and will yield more satisfactory and consistent results than will the conventional macro methods. No theory is included, and the procedures do not require the use of H_2S. An altogether practical laboratory outline.

N. W. R.

Annual Index—Journal of Chemical Education

VOLUME 23, 1946

The annual index for Volume 23 of the JOURNAL OF CHEMICAL EDUCATION is divided into two parts—an Author Index and a Subject Index, each alphabetically arranged. Letters (Ar), (B), and (C) have been used after the various items to designate, respectively: article, book review, and correspondence. The sections *Out of the Editor's Basket* and *What's Been Going On* are not indexed as to subject material, but contributors to these sections have been listed separately at the end of the Author Index.

AUTHOR INDEX

ABBOTT, CYRIL E. If I had a son (Ar). 438
ABRAHAMS, HAROLD J., AND ROBERT C. CROSSON, JR. Colloidal metals in non-aqueous solvents by the Bredig method (Ar). 289
ALEXANDER, A. E. Rubies and sapphires: Genuine and synthetic (Ar). 418
ALEXANDER, JEROME, *Editor*. Colloid chemistry: Theoretical and applied. Volume VI (B). 520
ALYEA, HUBERT N.—*See* BROOKES, VINCENT J.
AMSDEN, JOHN PAGE. Physical chemistry for premedical students (B). 362
ANSCHÜTZ, RICHARD—*See* VON RICHTER, VICTOR.
APFELBAUM, PERCY M.—*See* LOWY, ALEXANDER.
ARCHIBALD, J. G. The application of chemistry to animal husbandry (Ar). 49
ARDIS, ALAN—*See* GRUMMITT, OLIVER.
ASENJO, CONRADO F. Vicente Marcano (1848–1891): A pioneer chemist of Venezuela (Ar). 145
AUDRIETH, L. F. (C). 47

BACHMANN, WERNER E., *Editor*. Organic syntheses (B). 103
BAILAR, JOHN C.—*See* HOPKINS, B. SMITH.
BAILEY, C. F.—*See* CASEY, R. S.
BAKER, ELTON M. Quantitative analysis for premedical students (Ar). 567
BARNES, W. C. E. Operating an abstracting and information service within an industrial organization (Ar). 152
BARRON, HARRY. Modern plastics (B). 363
BATEMAN, J. B.—*See* HOBER, RUDOLPH.
BAYNE, HELEN (C). 348
BELCHER, RONALD, AND CECIL L. WILSON. Qualitative inorganic microanalysis (B). 624
BENDER, PAUL. Measurement of dipole moments (Ar). 179
BENNETT, GEORGE W. A navy V-12 course in quantitative analysis (Ar). 247
BERRY, A. J. Modern chemistry (B). 624
BICK, I. R. C. A simple laboratory hot-air dryer (Ar). 127
BINDER, L. O. Some small-scale organic preparations (Ar). 285
BLOCK, RICHARD J., AND DIANA BOLLING. The amino acid composition of proteins and foods (B). 364
BOLLING, DIANA—*See* BLOCK, RICHARD J.
BONNER, WILLIAM A. A versatile microscope hot stage (Ar). 601
BOWMAN, R. E. (C). 47
BRASTED, ROBERT C. Analysis of sodium nitrite by sulfamic acid: A gasometric analysis (Ar). 320
BREDIG, MAX A. (C). 46
BREWER, GEORGE E. F., AND MARY MARGARET TENBUSCH. The qualitative analysis of group III: A survey (Ar). 66
BRODY, SAMUEL. Bioenergetics and growth (B). 208
BROOKES, VINCENT J., AND HUBERT N. ALYEA. Poisons (B). 572
BUHLE, EMMETT L.—*See* WISEBLOGLE, F. Y.
BUNN, C. W. Chemical crystallography (B). 311
BURGER, CHARLES A.—*See* MAGLIO, M. MARTIN.
BURKE, WILLIAM R. (C). 309
BURT, C. PAULINE. A survey of synthetic medicinals (Ar). 358, 412
BURTON, E. F., AND W. H. KOHL. The electron microscope (B). 310

CABLE, EMMETT JAMES, ROBERT WARD GETCHELL, AND WILLIAM HENRY KADESCH. Science in a changing world (B). 312
CADBURY, W. E., JR.—*See* MELDRUM, W. B.
CALDWELL, W. T., AND WILLIAM ROGERS, JR. Chemistry in the premedical curriculum: Viewpoint of the college of liberal arts (Ar). 334
CALEY, EARLE R. Ancient Greek pigments (Ar). 314
CALKINS, F. C. Simple devices for measuring out small quantities of liquids (Ar). 604
CALVIN, D. BAILEY, AND B. M. HENDRIX. Chemistry in the premedical curriculum: The viewpoint of the medical college (Ar). 326
CAMPBELL, J. A. Atomic size and the periodic table (Ar). 525
Catalysis and the elementary chemistry course (Ar). 582
CARNEY, R. J.—*See* MELOCHE, C. C.
CASEY, R. S., C. F. BAILEY, AND G. J. COX. Punch card techniques and applications (Ar). 495
CASSARETTO, F. P.—*See* POTEMPA, S. J.
CASSIDY, HAROLD G. Methods of separation and tests for purity (Ar). 427
CHAMLIN, GEORGE R. The chemistry of benzene hexachloride and its insecticidal properties (Ar). 283
CHIOCCA, JOHN, AND ROBERT VAN REEN. SARA BENT RANSOM, *Editor*. Consumer chemistry (B). 104
COFFIN, C. C. A presentation of the thermodynamic functions (Ar). 584
COLE, JAMES W. Elastic silica: A demonstration (Ar). 97
COLLINS, EDWARD M., AND ROBERT W. SHELTON. A Kjeldahl determination of protein for general chemistry (Ar). 549
CONSIDINE, DOUGLAS M., AND DONALD P. ECKMAN. The basis of improved electronics (Ar). 274
COONS, K. W.—*See* YOUNG, C. B. F.
CORYELL, C. D. The scientific importance of the nuclear power projects (Ar). 395
COURTNEY, ROBERT P. A brief review of plastics chemistry (Ar). 306
COX, G. J.—*See* CASEY, R. S.
CRAGG, L. H. (C). 205
CRANE, E. J. Wartime scientific periodicals (Ar). 482
CROSSON, ROBERT C., JR.—*See* ABRAHAMS, HAROLD J.

DE MENT, JACK. Fluorochemistry (B). 260
The fluorochemistry of uranium (Ar). 213
DEMING, HORACE G. A device for teaching chemical arithmetic (Ar). 259
DEWITT, CHARLES B., AND L. B. ROGERS. Effective training in quantitative analysis (Ar). 169
DICKISON, RAYMOND R. Let your librarian look it up (Ar). 175
DIXON, MALCOLM. Manometric methods (B). 104
DRUCE, GERALD (C). 467

EASTWOOD, L. W. Gas in light alloys (B). 624
EBERHARDT, W. H. A demonstration mass spectrometer (Ar). 220
ECKMAN, DONALD P.—*See* CONSIDINE, DOUGLAS M.
EGLOFF, G. Physical constants of hydrocarbons (B). 468
EIDINOFF, MAXWELL L. Uranium fission: Discoveries leading to a chain reaction (Ar). 60

FAIRES, V. M.—*See* GRINTER, L. E.
FEIGL, FRITZ (C). 205
FELLNER, I. J. Patentability of organic compounds (Ar). 555
FENN, WALLACE O.—*See* HOBER, RUDOLPH.
FERGUSON, LLOYD N. Balancing equations for organic oxidation-reduction reactions (Ar). 550
FISHER, B. T.—*See* STERNBERG, H. W.
FOSTER, L. S. (C). 602
FRIER, W. T., AND ALBERT C. HOLLER. Introduction to industrial chemistry (B). 52
FROMM, F. (C). 309

GECKLER, ROBERT P.—*See* SUTTLE, JOHN F.
GETCHELL, ROBERT WARD—*See* CABLE, EMMETT JAMES.
GILLIS, DOROTHY—*See* ZAHND, HUGO.
GODDARD, DAVID R.—*See* HOBER, RUDOLPH.
GRANT, JULIUS. *Reviser and editor*. Hackh's chemical dictionary (B). 260
Editor. FRITZ PREGL. Quantitative organic microanalysis (B). 468
GRINTER, L. E., HARRY N. HOLMES, H. C. SPENCER, RUFUS OLDENBURGER, CHARLES HARRIS, R. G. KLOEFFLER, AND V. M. FAIRES. Engineering preview (B). 260
GRUMMITT, OLIVER, AND ALAN ARDIS. Reactions of sulfur dioxide with unsaturated hydrocarbons (Ar). 73
GULL, CLOYD DAKE. A punched card method for the bibliography, abstracting, and indexing of chemical literature (Ar). 500

HACKERMAN, NORMAN. The equilibrium concept in beginning college chemistry courses (Ar). 45
HALL, WILLIAM T. (C). 516
HARDEN, FLORENCE. Foreign technical publications (Ar). 489
HARRIS, CHARLES—*See* GRINTER, L. E.
HARROW, BENJAMIN—*See* LOWY, ALEXANDER.
HARTNELL, ELINOR D.—*See* WISEBLOGLE, F. Y.
HARVEY, H. W. Recent advances in the chemistry and biology of sea water (B). 206
HAUSER, ERNST A. (C). 46
HAUSMAN, HAROLD. Silicon organics (Ar). 16
HAWLEY, GESSNER G., AND SIGMUND W. LEIFSON. Atomic energy in war and peace (B). 207
HAZLEHURST, THOMAS H. How to solve problems and influence students (Ar). 445
HEITLER, W. Elementary wave mechanics (B). 416
HELBIG, W. A. Activated carbon (Ar). 98
HELDMAN, JULIUS D. Techniques of glass manipulation in scientific research (B). 472
HENDRIX, B. M.—*See* CALVIN D. BAILEY.
HERRINGTON, B. L. (C). 89
HILDEBRAND, JOEL H. Catalyzing the approach to equilibrium (Ar). 589
HILL, NORMAN C. The technical library of tomorrow (Ar). 474
HITCHCOCK, DAVID I.—*See* HOBER, RUDOLPH.
HOBER, RUDOLPH, DAVID I. HITCHCOCK, J. B. BATEMAN, DAVID R. GODDARD, AND WALLACE O. FENN. Physical chemistry of cells and tissues (B). 312
HOLLER, ALBERT C.—*See* FRIER, W. T.
HOLMES, HARRY N.—*See* GRINTER, L. E.
HOPKINS, B. SMITH, AND JOHN C. BAILAR. Essentials of general chemistry (B). 310
HORTON, WILLIAM S. On heats of solution (Ar). 393
HUTCHINSON, REV. GERALD F., S.J. (C). 89

IGNATIA, SISTER M., I.H.M. Science seminars (Ar). 149
ISOM, LANGLEY W. The application of colloid chemistry to the manufacture of inflated articles (Ar). 620

JACOBSON, C. A., *Compiler and Editor*. Encyclopedia of chemical reactions (B). 260

JAMES, T. H. Recent hypotheses concerning the mechanism of photographic development (Ar) ... 595
JONES, F. L. Nonsilica glass systems (Ar). 608

KADESCH, WILLIAM HENRY—*See* CABLE, EMMETT JAMES.
KAMERLING, SAMUEL E. Leaf pigments (Ar) ... 202
KAYE, JOSEPH—*See* KEENAN, JOSEPH H.
KEENAN, JOSEPH H., AND JOSEPH KAYE. Thermodynamic properties of air (B). 103
KEIGHTON, WALTER B. (C) ... 155
The objectives of premedical education: Viewpoint of the liberal arts college (Ar) ... 322
KENDALL, JAMES. Barbituric acid (Ar).. 2
KLEINBERG, JACOB. The positive character of the halogens (Ar) ... 559
KLICKSTEIN, HERBERT S., AND HENRY M. LEICESTER. Max Abramovich Blokh: His contributions to the history of chemistry (Ar) ... 451
KLOEFFLER, R. G.—*See* GRINTER, L. E.
KOHL, W. H.—*See* BURTON, E. F.

LAMBERT, RAYMOND H. Instrumentation in chemical analysis (Ar).. 610
LAUFFER, MAX A. Influenza virus (Ar).. 530
LEFFINGWELL, GEORGIA, AND MILTON LESSER. Soap in industry (B) ... 311
LEICESTER, HENRY M.—*See* KLICKSTEIN, HERBERT S.
LEIPSON, SIGMUND W.—*See* HAWLEY, GESSNER G.
LEMAY, PIERRE—*See* OESPER, RALPH E.
LESSER, MILTON—*See* LEFFINGWELL, GEORGIA.
LEVIN, IRVIN. The phase rule and topology (Ar) ... 183
LEVIN, JACOB. Nonferrous metals aid in steel's use (Ar) ... 142
LEVY, HYMAN, AND E. E. PREIDEL. Elementary statistics (B) ... 104
LIEBHAFSKY, H. A. Inner complex ions: A chelate species (Ar) ... 341
LOGUE, PAUL. Industrial uses of phosphates (Ar) ... 529
LOHSE, HENRY WILLIAM. Catalytic chemistry (B) ... 208
LOWY, ALEXANDER, AND BENJAMIN HARROW. *Revised by* PERCY M. APFELBAUM. Introduction to organic chemistry (B) ... 206
LUDER, W. F. The semantics of thermodynamics (Ar)
Part I ... 54
Part II ... 110

MACRAE, DUNCAN. The introduction to thermodynamics (Ar) ... 366
MAGLIO, M. MARTIN, AND CHARLES A. BURGER. Condensation reactions: A laboratory preparation of ethylidene glycerol (Ar) ... 174
MANNING, JOSEPH F.—*See* MASON, J. PHILLIP.
MARK, H., AND G. S. WHITBY, *Editors*. Advances in colloid science (B) ... 572
MARTELL, ARTHUR E. Entropy and the second law of thermodynamics (Ar).. 166
MASON, J. PHILLIP, AND JOSEPH F. MANNING. The technology of plastics and resins (B) ... 156
MCALPINE, R. K. Anomalous behavior of some oxidizing agents (Ar) ... 301
Percentage composition and the atomic theory (Ar) ... 227
Sensitivity of precipitation reactions of dilute electrolytes (Ar) ... 28
Separation and identification of cobalt, nickel, zinc, and manganese (Ar) ... 298
MCCULLOCH, LEON. Changing phenolphthalein from colorless to pink by adding acids (Ar) ... 473
Demonstration of combustion by catalysis (Ar) ... 524
MCELVAIN, SAMUEL M. The characterization of organic compounds (B) ... 156
MCGAVOCK, WILLIAM C. Organic oxidation-reduction-reactions (B) ... 103
MEE, A. J.—*See* VON RICHTER, VICTOR.
MELDRUM, W. B., AND W. E. CADBURY, JR. The cultural values of the sciences in the premedical program (Ar) ... 338
MELLON, M. G. Colorimetry for chemists (B) ... 260
MELOCHE, C. C., AND R. J. CARNEY. Illustration and demonstration in elementary volumetric analysis (Ar) ... 198
MERRITT, LYNNE L., JR.—*See* SUTTLE, JOHN F.
MICHAELIS, L. Demonstration of the existence of a free radical (Ar) ... 317
MILLS, JOHN. Electronics: Today and tomorrow (B) ... 155
MINTER, CLARKE C. The thermal conductivity method of gas analysis (Ar). 237
MOELLER, THERALD. Inorganic nitrosyl compounds (Ar).
I. Cationic and anionic compounds... 441
II. Coordination compounds ... 542
MOORE, ALEXANDER M.—*See* WISELOGLE, F. Y.
MOOS, GILBERT E. Laboratory acid cleaning bath (Ar) ... 465
MOUQUIN, HENRY—*See* SCHAUM, DANIEL.

NOYES, RICHARD M. Science teachers and the atomic bomb (Ar) ... 343

OELKE, W. C. Photography as a basic science (Ar) ... 593
OESPER, RALPH E. Friedrich Fichter (Ar). 394
Paul Karrer (Ar) ... 392
—AND PIERRE LEMAY. Claude Louis Berthollet (Ar) ... 158, 230
OLDENBURGER, RUFUS—*See* GRINTER, L. E.
OOMS, CASPER W. The American patent system: Its relation to technical library techniques (Ar) ... 478

PALIT, SANTI R. Solvent action and "co-solvency": A lecture demonstration (Ar) ... 182
PARODI, JOHN—*See* SELWOOD, P. W.
PATTERSON, AUSTIN M. Wartime chemical education through E.S.M.W.T. (Ar). 463
PERRY, JAMES W. Chemical Russian, self-taught (Ar).
II. The vocabulary problem ... 22
III. Russian inorganic chemical nomenclature ... 116
Errata ... 248
PHILLIPS, RALPH W., AND GRANT VAN HUYSEN. The chemistry of tooth tissues (Ar) ... 579
PICKARD, R. FORTESCUE. Time, number, and the atom (B) ... 260
PITHA, JOHN. A simple sublimation apparatus (Ar) ... 403
POND, FRANCIS J.—*See* SCHAUM, DANIEL.
PORTER, LYMAN E. The semimicro detection of thiocyanate and halide ions (Ar) ... 402
POTEMPA, S. J., AND F. P. CASSARETTO. A condenser-receiver unit (Ar) ... 219
POZZI-ESCOT, M.-EMM (C) ... 205
PREGL, FRITZ—*See* GRANT, JULIUS.
PREIDEL, E. E.—*See* LEVY, HYMAN.
PRINGSHEIM, P., AND M. VOGEL. Luminescence of liquids and solids (B) ... 156

RABINOWITCH, EUGENE I. Photosynthesis and related processes. Volume I (B) ... 52
RANSOM, SARA BENT—*See* CHIOCCA, JOHN.
RICE, C. MABEL. Dictionary of geological terms (B) ... 104
ROBERTS, SIDNEY H. Chemistry and a life insurance company (Ar) ... 568
ROGERS, L. B.—*See* DEWITT, CHARLES B.
ROGERS, WILLIAM, JR.—*See* CALDWELL, W. T.

SACHANEN, A. N. The chemical constituents of petroleum (B) ... 416
SAMPEY, JOHN R. A chemist views Africa (Ar) ... 318
Selecting a graduate school for training in chemistry (Ar) ... 583
SAUL, EVERETT L. Odor and the organic chemist (Ar) ... 296
SCHAUM, DANIEL, *edited and revised by* HENRY MOUQUIN AND WILLIAM J. WISWESSER, *with problems by* FRANCIS J. POND. Theory and problems for students of college chemistry (B) ... 207
SCHOOLEY, DAISY L. Peace demands for aluminum expand (Ar) ... 399
SCHUELER, FRED W. New interest in the nitrogen halides (Ar) ... 168
SCHULZE, ELSE L. Wanted: More library chemists (Ar) ... 176
SEIFFERT, GUSTAV. Virus diseases in man, animal, and plant (B) ... 155
SELWOOD, P. W., AND JOHN PARODI. Magnetic anisotropy of organic solids (Ar) ... 574
SEMAT, HENRY. Introduction to atomic physics (B) ... 624
SETTERFIELD, H. E. Scientific and humanistic objectives in premedical education: Viewpoint of the medical college (Ar) ... 330
SHELTON, ROBERT W.—*See* COLLINS, EDWARD M.
SIDDONS, GEORGE F.—*See* SUTTLE, JOHN F.
SILVERMAN, ALEXANDER. Graduate assistants: Their stipends (Ar) ... 351
SIMONDS, HERBERT R. Industrial plastics (B) ... 520
SINNOTT, EDMUND W. Plants hold the basic patents (Ar) ... 35
SISTER MARIE CLOTILDE. Teaching total acidity (Ar) ... 433
SLATE, FLOYD O. Organization of the technical report (Ar) ... 439
Training for technical report writing (Ar) ... 398
SMALLWOOD, HUGH M. Statistical methods of research (Ar) ... 352
SORUM, C. H. Problems for general chemistry and qualitative analysis (B) ... 104
SOUTHGATE, THERESE. A chemist's prayer (Ar) ... 507
SPACHT, RONALD B. Electrolytic control of corrosion (Ar) ... 195
The corrosion resistance of aluminum and its alloys (Ar) ... 253
SPENCER, H. C.—*See* GRINTER, L. E.
SQUIRES, A. M. The fractionation of isotopes (Ar) ... 538
STANDEN, ANTHONY. Why do we believe in electrons? (Ar) ... 356
STEACIE, E. W. R. Atomic and free radical reactions (B) ... 362
STEMPEL, GUIDO H., JR. Rearrangement of substituted benzopinacols: Application of electronic theories (Ar) ... 434
STERNBERG, H. W., AND B. T. FISHER. A condensed water collector (Ar) ... 397
STETTEN, DEWITT, JR. Scientific values of the humanities in the premedical program (Ar) ... 336
STEVENSON, CHARLES A. Note on van der Waal's reduced equation of state (Ar). 148
STONE, CHARLES H. Crystallization (Ar). 404
Nitrites (Ar) ... 196
Selenium (Ar) ... 349
Youthful Faradays (Ar) ... 249
STRAIN, HAROLD H. Indispensable leaf yellow (Ar) ... 262
SUTTLE, JOHN F., AND ROBERT P. GECKLER. An apparatus for the preparation of tellurium tetrachloride (Ar) ... 135
SUTTLE, JOHN F., LYNNE L. MERRITT, JR., AND GEORGE F. SIDDONS. Conversion of a bomb-release control to activate a solenoid stirrer (Ar) ... 612
SUTTON, PAUL PORTER. Conversion factors in physical chemistry (Ar) ... 126
SWEATT, H. B. The properties of animal glue (Ar) ... 192

TAFT, ROBERT, JR.—*See* VANDERWERF, CALVIN A.
TENBUSCH, MARY MARGARET—*See* BREWER, GEORGE E. F.
THEILHEIMER, W. Synthetische methoden der organischen chemie. Repertorium I (B) ... 310
THIESSEN, G. W. Use of the neon bulb for conductivity demonstration (Ar) ... 267
THOMPSON, ELIZABETH. Salt shaker wedding (Ar) ... 350
TOLPIN, J. G. Russian in the chemist's curriculum (Ar) ... 123
Searching the Russian technical literature (Ar) ... 485
TWISS, SUMNER B. Advancing fronts in chemistry: High polymers (B) ... 207

VAN HUYSEN, GRANT—*See* PHILLIPS, RALPH W.
VAN KLOOSTER, H. S. Franklin and Lavoisier (Ar) ... 107
The Lavoisier bicentenary exposition in Paris (Ar) ... 210
VAN REEN, ROBERT—*See* CHIOCCA, JOHN.
VANDERWERF, CALVIN A., AND ROBERT TAFT, JR. Construction of a fractionating column (Ar) ... 82
VERDOORN, FRANS. Relief packages for scientific workers in western Europe (C) 246
VERNON, ARTHUR A. Particle size determination (Ar) ... 448
VOGEL, M.—*See* PRINGSHEIM, P.
VON RICHTER, VICTOR. RICHARD ANSCHÜTZ, *Editor*. A. J. MEE, *Translator*. Chemistry of the carbon compounds, The. Volume III (B) ... 520

WAKEHAM, G. Note on teaching the metric system (Ar) ... 134
Teaching valence in beginning chemistry (Ar) ... 43
WALTON, H. F. Ion exchange (Ar) ... 454
WASLEY, W. L. The objectives of qualitative analysis (Ar) ... 357
WEISSMAN, SIMON A. The anomalous behavior of liquid helium II (Ar) ... 223
WENDLAND, RAY T. A system of characterization of pure hydrocarbons; Refractometric analysis as a key to the evaluation of structure (Ar) ... 3
WHITBY, G. S.—*See* MARK, H.
WILLIAMS, RUSSELL R., JR. Nuclear energetics (Ar) ... 508
Nuclear reactions (Ar) ... 423
WILSON, CECIL L.—*See* BELCHER, RONALD.
WILSON, VELMA. An inventory form for a small laboratory (Ar) ... 165
WISELOGLE, F. Y., EMMETT L. BUHLE,

ELINOR D. HARTNELL, ALEXANDER M. MOORE, AND LOUISE R. WISELOGLE. A new system for the classification of compounds: A contribution from the survey of antimalarial drugs (Ar)..... 375
WISELOGLE, LOUISE R.—*See* WISELOGLE, F. Y.
WISWESSER, WILLIAM J. Invitation to chemistry: R.S.V.P. (Ar)..... 271
—*See* SCHAUM, DANIEL.
WRIGHT, CLARENCE W. George Washington Carver (Ar)..... 268
WRIGHT, R. H. Faraday and the radio engineer (Ar)..... 251
YOUNG, C. B. F., AND K. W. COONS. Surface active agents (B)..... 363
ZAHND, HUGO, AND DOROTHY GILLIS. Chemical knowledge in the New Testament (Ar)..... 90

OUT OF THE EDITOR'S BASKET
BUTCHER, B. C..... 515
ESSENBERG, J. M..... 565
WENDER, SIMON H..... 243

WHAT'S BEEN GOING ON
DEGERING, ED. F..... 20

SUBJECT INDEX

ABRAMOVICH BOLKH, MAX: His contributions to the history of chemistry. HERBERT S. KLICKSTEIN AND HENRY M. LEICESTER..... 451
Abstracting and information service within an industrial organization, Operating an. W. C. E. BARNES..... 152
Abstracting, bibliography, and indexing of chemical literature, A punched card method for the. CLOYD DAKE GULL. 500
Acid, Barbituric. JAMES KENDALL..... 2
Acid cleaning bath, Laboratory. GILBERT E. MOOS..... 465
Acid, sulfamic, Analysis of sodium nitrite by: A gasometric analysis. ROBERT C. BRASTED..... 320
Acidity, total, Teaching. SISTER MARIE CLOTILDE..... 433
Acids, Changing phenolphthalein from colorless to pink by adding. LEON MCCULLOCH..... 473
Action, Solvent, and "co-solvency": A lecture demonstration. SANTI R. PALIT..... 182
Activated carbon. W. A. HELBIG..... 98
Active and Light..... 444
Ad astra per aspera..... 36
Adding acids, Changing phenolphthalein from colorless to pink by. LEON MCCULLOCH..... 473
Advances, Wartime, in steel technology... 470
Africa, A chemist views. JOHN R. SAMPEY. 318
Agents, oxidizing, Anomalous behavior of some. R. K. MCALPINE..... 301
Airy topic, An..... 205
Alchemy, Plant..... 44
Alloys, The corrosion resistance of aluminum and its. RONALD B. SPACHT.... 253
Aluminum and its alloys, The corrosion resistance of. RONALD B. SPACHT..... 253
Aluminum, Peace demands for, expand. DAISY L. SCHOOLEY..... 399
American Chemical Society, Division of chemical education of the..... 618
American patent system, The: Its relation to technical library techniques. CASPER W. OOMS..... 478
American scientist, An: George Washington Carver. CLARENCE W. WRIGHT..... 268
Analysis, A gasometric: Analysis of sodium nitrite by sulfamic acid. ROBERT C. BRASTED..... 320
Analysis, chemical, Instrumentation in. RAYMOND H. LAMBERT..... 610
Analysis, elementary volumetric, Illustration and demonstration in. C. C. MELOCHE AND R. J. CARNEY..... 198
Analysis, gas, The thermal conductivity method of. CLARKE C. MINTER..... 237
Analysis of sodium nitrite by sulfamic acid: A gasometric analysis. ROBERT C. BRASTED..... 320
Analysis, qualitative, The objectives of. W. L. WASLEY..... 357
Analysis, quantitative, A navy V-12 course in. GEORGE W. BENNETT..... 247
Analysis, quantitative, Effective training in. CHARLES B. DEWITT AND L. B. ROGERS 169
Analysis, Quantitative, for premedical students. ELTON M. BAKER..... 567
Analysis, Refractometric, as a key to the evaluation of structure: A system of characterization of pure hydrocarbons. RAY T. WENDLAND..... 3
Analysis, The qualitative, of group III: A survey. MARY MARGARET TENBUSCH AND GEORGE E. F. BREWER... 66
Ancient Greek pigments. EARLE R. CALEY 314
Animal glue, The properties of. H. B. SWEATT..... 192
Animal husbandry, The application of chemistry to. J. G. ARCHIBALD..... 49
Anionic and Cationic compounds: I, Inorganic nitrosyl compounds. THERALD MOELLER..... 441
Anisotropy, Magnetic, of organic solids. P. W. SELWOOD AND JOHN PARODI.... 574
Anomalous behavior of liquid helium II, The. SIMON A. WEISSMAN..... 223
Anomalous behavior of some oxidizing agents. R. K. MCALPINE..... 301
Antimalarial drugs, A contribution from the survey of: A new system for the classification of compounds. F. Y. WISELOGLE ET AL..... 375
Apparatus, A simple sublimation. JOHN PITHA..... 403
Apparatus for the preparation of tellurium tetrachloride, An. JOHN F. SUTTLE AND ROBERT P. GECKLER..... 135
Application of chemistry to animal husbandry, The. J. G. ARCHIBALD..... 49
Application of colloid chemistry to the manufacture of inflated articles, The. LANGLEY W. ISOM..... 620
Application of electronic theories: Rearrangement of substituted benzopinacols. GUIDO H. STEMPEL, JR..... 434
Applications, Punch card techniques and, R. S. CASEY, C. F. BAILEY, AND G. J. COX..... 495
Approach to equilibrium, Catalyzing the. JOEL H. HILDEBRAND..... 589
Arithmetic, chemical, A device for teaching. HORACE G. DEMING..... 259
Articles, inflated, The application of colloid chemistry to the manufacture of. LANGLEY W. ISOM..... 620
Arts, liberal, Viewpoint of the college of: Chemistry in the premedical curriculum. W. T. CALDWELL AND WILLIAM ROGERS, JR..... 334
Aspera, Ad astra per..... 36
Assistants, Graduate: Their stipends. ALEXANDER SILVERMAN..... 351
Astra, Ad, per aspera..... 36
Atomic bomb, Science teachers and the. RICHARD M. NOYES..... 343
Atomic size and the periodic table. J. A. CAMPBELL..... 525
Atomic theory, Percentage composition and the. R. K. MCALPINE..... 227
Authors, Notice to..... 636

BALANCING equations for organic oxidation-reduction reactions. LLOYD N. FERGUSON..... 550
Barbituric acid. JAMES KENDALL..... 2
Basic patents, Plants hold the. EDMUND W. SINNOTT..... 35
Basic science, Photography as a. W. C. OELKE..... 593
Basis of improved electronics, The. DOUGLAS M. CONSIDINE AND DONALD P. ECKMAN..... 274
Bath, Laboratory acid cleaning. GILBERT E. MOOS..... 465
Beginning chemistry, Teaching valence in. G. WAKEHAM..... 43
Beginning college chemistry courses, The equilibrium concept in. NORMAN HACKERMAN..... 45
Behavior, Anomalous, of some oxidizing agents. R. K. MCALPINE..... 301
Behavior, The anomalous, of liquid helium II. SIMON A. WEISSMAN..... 223
Benzene hexachloride, The chemistry of, and its insecticidal properties. GEORGE R. CHAMLIN..... 283
Benzopinacols, substituted, Rearrangement of: Application of electronic theories. GUIDO H. STEMPEL, JR..... 434
Berthollet, Claude Louis. PIERRE LEMAY AND RALPH E. OESPER..... 158, 230
Bibliography, abstracting, and indexing of chemical literature, A punched card method for the. CLOYD DAKE GULL.. 500
Bicentenary Lavoisier exposition in Paris, The. H. S. VAN KLOOSTER..... 210
Blokh, Max Abramovich: His contributions to the history of chemistry. HERBERT S. KLICKSTEIN AND HENRY M. LEICESTER..... 451
Bomb, atomic, Science teachers and the. RICHARD M. NOYES..... 343
Bomb-release control, Conversion of a, to activate a solenoid stirrer. JOHN F. SUTTLE, LYNNE L. MERRITT, JR., AND GEORGE F. SIDDONS..... 612
Books—War victims..... 312
Bredig method, Colloidal metals in nonaqueous solvents by the. ROBERT C. CROSSON, JR., AND HAROLD J. ABRAHAMS..... 289
Brief review of plastics chemistry, A. ROBERT P. COURTNEY..... 306
Bulb, Use of the neon, for conductivity demonstration. G. W. THIESSEN..... 267

Books reviewed:

Acid, The amino, composition of proteins and foods. RICHARD J. BLOCK AND DIANA BOLLING..... 364
Active agents, Surface. C. B. F. YOUNG AND K. W. COONS..... 363
Advances in colloid science. Volume II. *Edited by* H. MARK AND G. S. WHITBY. 572
Advances, Recent, in the chemistry and biology of sea water. H. W. HARVEY. 206
Advancing fronts in chemistry: High polymers. SUMNER B. TWISS..... 207
Agents, Surface active. C. B. F. YOUNG AND K. W. COONS..... 363
Air, Thermodynamic properties of. JOSEPH H. KEENAN AND JOSEPH KAYE..... 103
Alloys, light, Gas in. L. W. EASTWOOD. 624
Amino acid composition of proteins and foods, The. RICHARD J. BLOCK AND DIANA BOLLING..... 364
Analysis, Organic reagents for organic... 311
Analysis, qualitative, Problems for general chemistry and. C. H. SORUM. 104
Animal, man, and plant, Virus diseases in. GUSTAV SEIFFERT..... 155
Atom, Time and number. R. FORTESCUE PICKARD..... 260
Atomic and free radical reactions. E. W. R. STEACIE..... 362
Atomic energy in war and peace. GESSNER G. HAWLEY AND SIGMUND W. LEIFSON..... 207
Atomic physics, Introduction to. HENRY SEMAT..... 636
Bioenergetics and growth. SAMUEL BRODY..... 208
Biology and chemistry of sea water, Recent advances in the. H. W. HARVEY..... 206
Carbon compounds, The chemistry of the. Volume III. VICTOR VON RICHTER. *Edited by* RICHARD ANSCHÜTZ. *Translated by* A. J. MEE..... 520
Catalytic chemistry. HENRY WILLIAM LOHSE..... 208
Cells and tissues, Physical chemistry of. RUDOLPH HOBER, DAVID I. HITCHCOCK, J. B. BATEMAN, DAVID R. GODDARD, AND WALLACE O. FENN.... 312
Changing world, Science in a. EMMETT JAMES CABLE, ROBERT WARD GETCHELL, AND WILLIAM HENRY KADESCH 312
Characterization of organic compounds, The. SAMUEL M. MCELVAIN..... 156
Chemical constituents of petroleum, The. A. N. SACHANEN..... 416
Chemical crystallography. C. W. BUNN 311
Chemical dictionary, Hackh's. *Revised and edited by* JULIUS GRANT..... 260
Chemical reactions, Encyclopedia of. *Compiled and edited by* C. A. JACOBSON. 260
Chemie, organischen, Synthetische methoden der. Repertorium I. W. THEILHEIMER..... 310
Chemistry, Advancing fronts in: High polymers. SUMNER B. TWISS..... 207
Chemistry and biology of sea water, Recent advances in the. H. W. HARVEY..... 206

Chemistry, Catalytic. HENRY WILLIAM LOHSE..... 208
Chemistry, college, Theory and problems for students of. DANIEL SCHAUM..... 207
Chemistry, Colloid: Theoretical and applied. Volume VI. *Edited by* JEROME ALEXANDER..... 520
Chemistry, Consumer. JOHN CHIOCCA AND ROBERT VAN REEN. *Edited by* SARA BENT RANSOM..... 104
Chemistry, general, and qualitative analysis, Problems for. C. H. SORUM. 104
Chemistry, general, Essentials of. B. SMITH HOPKINS AND JOHN C. BAILAR. 310
Chemistry, industrial, Introduction to. W. T. FRIER AND ALBERT C. HOLLER. 52
Chemistry, Modern. A. J. BERRY..... 624
Chemistry of the carbon compounds. Volume III. VICTOR VON RICHTER. *Edited by* RICHARD ANSCHÜTZ. *Translated by* A. J. MEE..... 520
Chemistry, organic, Introduction to. ALEXANDER LOWY AND BENJAMIN HARROW, *revised by* PERCY M. APPELBAUM..... 206
Chemistry, Physical, for premedical students. JOHN PAGE AMSDEN..... 362
Chemistry, Physical, of cells and tissues. RUDOLPH HOBER, DAVID I. HITCHCOCK, J. B. BATEMAN, DAVID R. GODDARD, AND WALLACE O. FENN.... 312
Chemists, Colorimetry for. M. G. MELLON..... 260
College chemistry, Theory and problems for students of. DANIEL SCHAUM.... 207
Colloid chemistry: Theoretical and applied. Volume VI. *Edited by* JEROME ALEXANDER..... 520
Colloid science, Advances in. Volume II. *Edited by* H. MARK AND G. S. WHITBY. 572
Colorimetry for chemists. M. G. MELLON..... 260
Composition, The amino acid, of proteins and foods. RICHARD J. BLOCK AND DIANA BOLLING..... 364
Compounds, carbon, The chemistry of the. Volume III. VICTOR VON RICHTER. *Edited by* RICHARD ANSCHÜTZ. *Translated by* A. J. MEE..... 520
Compounds, organic, The characterization of. SAMUEL M. MCELVAIN..... 156
Constants, Physical, of hydrocarbons. G. EGLOFF..... 468
Constituents, The chemical, of petroleum. A. N. SACHANEN..... 416
Consumer chemistry. JOHN CHIOCCA AND ROBERT VAN REEN. *Edited by* SARA BENT RANSOM..... 104
Crystallography, Chemical. C. W. BUNN 311
Dictionary, Hackh's chemical. *Revised and edited by* JULIUS GRANT..... 260
Dictionary of geological terms. C. MABEL RICE..... 104
Diseases, Virus, in man, animal, and plant. GUSTAV SEIFFERT..... 155
Electron microscope, The. E. F. BURTON AND W. H. KOHL..... 310
Electronics: Today and tomorrow. JOHN MILLS..... 155
Elementary statistics. HYMAN LEVY AND E. E. PREIDEL..... 104
Elementary wave mechanics. W. HEITLER..... 416
Encyclopedia of chemical reactions. *Compiled and edited by* C. A. JACOBSON. 260
Energy, Atomic, in war and peace. GESSNER G. HAWLEY AND SIGMUND W. LEIFSON..... 207
Engineering preview. L. E. GRINTER, HARRY N. HOLMES, H. C. SPENCER, RUFUS OLDENBURGER, CHARLES HARRIS, R. G. KLOEFFLER, AND V. M. FAIRES..... 260
Essentials of general chemistry. B. SMITH HOPKINS AND JOHN C. BAILAR. 310
Fluorochemistry. JACK DEMENT..... 260
Foods and proteins, The amino acid composition of. RICHARD J. BLOCK AND DIANA BOLLING..... 364
Free radical reactions, Atomic and. E. W. R. STEACIE..... 362
Fronts, Advancing, in chemistry: High polymers. SUMNER B. TWISS..... 207
Gas in light alloys. L. W. EASTWOOD... 624
General chemistry and qualitative analysis, Problems for. C. H. SORUM..... 104
General chemistry, Essentials of. B. SMITH HOPKINS AND JOHN C. BAILAR.. 310
Geological terms, Dictionary of. C. MABEL RICE..... 104
Glass manipulation, Techniques of, in scientific research. JULIUS D. HELDMAN..... 572
Growth and Bioenergetics. SAMUEL BRODY..... 208
Hackh's chemical dictionary. *Revised and edited by* JULIUS GRANT..... 260
High polymers: Advancing fronts in chemistry. SUMNER B. TWISS..... 207
Hydrocarbons, Physical constants of. G. EGLOFF..... 468
Industrial chemistry, Introduction to. W. T. FRIER AND ALBERT C. HOLLER. 52
Industrial plastics. HERBERT R. SIMONDS..... 520
Industry, Soap in. GEORGIA LEFFINGWELL AND MILTON LESSER..... 311
Inorganic microanalysis, Qualitative. RONALD BELCHER AND CECIL L. WILSON..... 636
Introduction to atomic physics. HENRY SEMAT..... 636
Introduction to industrial chemistry. W. T. FRIER AND ALBERT C. HOLLER. 52
Introduction to organic chemistry. ALEXANDER LOWY AND BENJAMIN HARROW, *revised by* PERCY M. APPELBAUM..... 206
Light alloys, Gas in. L. W. EASTWOOD. 624
Liquids and solids, Luminescence of. P. PRINGSHEIM AND M. VOGEL..... 156
Luminescence of liquids and solids. P. PRINGSHEIM AND M. VOGEL..... 156
Man, animal, and plant, Virus diseases in. GUSTAV SEIFFERT..... 155
Manipulation, glass, Techniques of, in scientific research. JULIUS D. HELDMAN..... 572
Manometric methods. MALCOLM DIXON 104
Mechanics, Elementary wave. W. HEITLER..... 416
Methoden, Synthetische, der organischen chemie. Repertorium I. W. THEILHEIMER..... 310
Methods, Manometric. MALCOLM DIXON 104
Microanalysis, Qualitative inorganic. RONALD BELCHER AND CECIL L. WILSON..... 624
Microanalysis, Quantitative organic. *Based on the methods of* FRITZ PREGL. *Revised and edited by* JULIUS GRANT... 468
Microscope, The electron. E. F. BURTON AND W. H. KOHL..... 310
Modern chemistry. A. J. BERRY..... 624
Modern plastics. HARRY BARRON..... 363
Number, Time, and the atom. R. FORTESCUE PICKARD..... 260
Organic chemistry, Introduction to. ALEXANDER LOWY AND BENJAMIN HARROW, *revised by* PERCY M. APPELBAUM..... 206
Organic compounds, The characterization of. SAMUEL M. MCELVAIN..... 156
Organic microanalysis, Quantitative. *Based on the methods of* FRITZ PREGL. *Revised and edited by* JULIUS GRANT... 468
Organic oxidation-reduction reactions. WILLIAM C. MCGAVOCK..... 103
Organic reagents for organic analysis.... 311
Organic syntheses. *Edited by* WERNER E. BACHMANN..... 103
Organischen chemie, Synthetische methoden der. Repertorium I. W. THEILHEIMER..... 310
Oxidation-reduction reactions, Organic. WILLIAM C. MCGAVOCK..... 103
Peace and war, Atomic energy in. GESSNER G. HAWLEY AND SIGMUND W. LEIFSON..... 207
Petroleum, The chemical constituents of. A. N. SACHANEN..... 416
Photosynthesis and related processes. EUGENE I. RABINOWITCH..... 52
Physical chemistry for premedical students. JOHN PAGE AMSDEN..... 362
Physical chemistry of cells and tissues. RUDOLPH HOBER, DAVID I. HITCHCOCK, J. B. BATEMAN, DAVID R. GODDARD, AND WALLACE O. FENN.... 312
Physical constants of hydrocarbons. G. EGLOFF..... 468
Physics, atomic, Introduction to. HENRY SEMAT..... 624
Plant, animal, and man, Virus diseases in. GUSTAV SEIFFERT..... 155
Plastics and resins, The technology of. J. PHILLIP MASON AND JOSEPH F. MANNING..... 156
Plastics, Industrial. HERBERT R. SIMONDS..... 520
Plastics, Modern. HARRY BARRON..... 363
Poisons. VINCENT J. BROOKES AND HUBERT N. ALYEA..... 572
Polymers, High: Advancing fronts in chemistry. SUMNER B. TWISS..... 207
Premedical students, Physical chemistry for. JOHN PAGE AMSDEN..... 362
Preview, Engineering. L. E. GRINTER, HARRY N. HOLMES, RUFUS OLDENBURGER, CHARLES HARRIS, R. G. KLOEFFLER, AND V. M. FAIRES..... 260
Problems and Theory for students of college chemistry. DANIEL SCHAUM. 207
Problems for general chemistry and qualitative analysis. C. H. SORUM... 104
Processes, related, and Photosynthesis. EUGENE I. RABINOWITCH..... 52
Properties, Thermodynamic, of air. JOSEPH H. KEENAN AND JOSEPH KAYE..... 103
Proteins and foods, The amino acid composition of. RICHARD J. BLOCK AND DIANA BOLLING..... 364
Qualitative analysis, Problems for general chemistry and. C. H. SORUM..... 104
Qualitative inorganic microanalysis. RONALD BELCHER AND CECIL L. WILSON..... 624
Quantitative organic microanalysis. *Based on the methods of* FRITZ PREGL. *Revised and edited by* JULIUS GRANT... 468
Radical reactions, free, Atomic and. E. W. R. STEACIE..... 362
Reactions, Atomic and free radical. E. W. R. STEACIE..... 362
Reactions, chemical, Encyclopedia of. *Compiled and edited by* C. A. JACOBSON. 260
Reactions, Organic oxidation-reduction. WILLIAM C. MCGAVOCK..... 103
Reagents, Organic, for organic analysis.. 311
Recent advances in the chemistry and biology of sea water. H. W. HARVEY. 206
Reduction-oxidation reactions, Organic. WILLIAM C. MCGAVOCK..... 103
Related processes and Photosynthesis. EUGENE I. RABINOWITCH..... 52
Research, scientific, Techniques of glass manipulation in. JULIUS D. HELDMAN. 572
Resins and plastics, The technology of. J. PHILLIP MASON AND JOSEPH F. MANNING..... 156
Science, colloid, Advances in. Volume II. *Edited by* H. MARK AND G. S. WHITBY. 572
Science in a changing world. EMMETT JAMES CABLE, ROBERT WARD GETCHELL, AND WILLIAM HENRY KADESCH. 312
Scientific research, Techniques of glass manipulation in. JULIUS D. HELDMAN..... 572
Sea water, Recent advances in the chemistry and biology of sea water. H. W. HARVEY..... 206
Soap in industry. GEORGIA LEFFINGWELL AND MILTON LESSER..... 311
Solids and liquids, Luminescence of. P. PRINGSHEIM AND M. VOGEL..... 156
Statistics, Elementary. HYMAN LEVY AND E. E. PREIDEL..... 104
Students of college chemistry, Theory and problems for. DANIEL SCHAUM..... 207
Students, premedical, Physical chemistry for. JOHN PAGE AMSDEN..... 362
Surface active agents. C. B. F. YOUNG AND K. W. COONS..... 363
Syntheses, Organic. *Edited by* WERNER E. BACHMANN..... 103
Synthetische methoden der organischen chemie. Repertorium I. W. THEILHEIMER..... 310
Techniques of glass manipulation in scientific research. JULIUS D. HELDMAN.. 572
Technology of plastics and resins, The. J. PHILLIP MASON AND JOSEPH F. MANNING..... 156
Terms, geological, Dictionary of. C. MABEL RICE..... 104
Theory and problems for students of college chemistry. DANIEL SCHAUM.. 207
Thermodynamic properties of air. JOSEPH H. KEENAN AND JOSEPH KAYE..... 103
Time, number and the atom. R. FORTESCUE PICKARD..... 260
Tissues and cells, Physical chemistry of. RUDOLPH HOBER, DAVID I. HITCHCOCK, J. B. BATEMAN, DAVID R. GODDARD, AND WALLACE O. FENN.... 312
Today and tomorrow: Electronics. JOHN MILLS..... 155
Tomorrow and Today: Electronics. JOHN MILLS..... 155
Virus diseases in man, animal, and plant. GUSTAV SEIFFERT..... 155
War and peace, Atomic energy in. GESSNER G. HAWLEY AND SIGMUND LEIFSON..... 207
Water, sea, Recent advances in the chemistry and biology of. H. W. HARVEY..... 206
Wave mechanics, Elementary. W. HEITLER..... 416
World, changing, Science in a. EMMETT JAMES CABLE, ROBERT WARD GETCHELL, AND WILLIAM HENRY KADESCH. 312

CARBON, Activated. W. A. HELBIG.... 98
Card method, A punched, for the bibliography, abstracting, and indexing of chemical literature. CLOYD DAKE GULL..... 500
Card techniques, Punch, and applications. R. S. CASEY, C. F. BAILEY, AND G. J. COX..... 495
Carver, George Washington: An American scientist. CLARENCE W. WRIGHT..... 268
Catalysis and the elementary chemistry course. J. A. CAMPBELL..... 582

Catalysis, Demonstration of combustion by. LEON MCCULLOCH 524
Catalyzing the approach to equilibrium. JOEL H. HILDEBRAND 589
Cationic and anionic compounds: I, Inorganic nitrosyl compounds. THERALD MOELLER 441
Chain reaction, Discoveries leading to a: Uranium fission. MAXWELL L. EIDINOFF 60
Changing phenolphthalein from colorless to pink by adding acids. LEON MCCULLOCH 473
Character, The positive, of the halogens. JACOB KLEINBERG 559
Characterization, A system of, of pure hydrocarbons: Refractometric analysis as a key to the evaluation of structure. RAY T. WENDLAND 3
Chelate species, A: Inner complex ions. H. A. LIEBHAFSKY 341
Chemical analysis, Instrumentation in. RAYMOND H. LAMBERT 610
Chemical arithmetic, A device for teaching. HORACE G. DEMING 259
Chemical education, Division of, of the American Chemical Society 618
Chemical education, Officers and committees of the division of 48
Chemical education, Wartime, through E.S.M.W.T. AUSTIN M. PATTERSON 463
Chemical knowledge in the New Testament. HUGO ZAHND AND DOROTHY GILLIS 90, 128
Chemical literature, A punched card method for the bibliography, abstracting, and indexing of. CLOYD DAKE GULL 500
Chemical microscopy at Cornell, Summer courses in 194
Chemical Russian, self-taught. JAMES W. PERRY.
II. The vocabulary problem 22
III. Russian inorganic chemical nomenclature 116
Errata 248
Chemical Society, American, Division of chemical education of the 618
Chemist, A pioneer, of Venezuela: Vicente Marcano (1848–1891). CONRADO F. ASENJO 145
Chemist, organic, Odor and the. EVERETT L. SAUL 296
Chemist views Africa, A. JOHN R. SAMPEY 318
Chemistry and a life insurance company. SIDNEY H. ROBERTS 568
Chemistry, beginning, Teaching valence in. G. WAKEHAM 43
Chemistry, colloid, The application of, to the manufacture of inflated articles. LANGLEY W. ISOM 620
Chemistry course, elementary, Catalysis and the. J. A. CAMPBELL 582
Chemistry courses, beginning college, The equilibrium concept in. NORMAN HACKERMAN 45
Chemistry, general, A Kjeldahl determination of protein for. EDWARD M. COLLINS AND ROBERT W. SHELTON 549
Chemistry, His contributions to the history of: Max Abramovich Blokh. HERBERT S. KLICKSTEIN AND HENRY M. LEICESTER 451
Chemistry in the premedical curriculum: The viewpoint of the medical college. D. BAILEY CALVIN AND B. M. HENDRIX. 326
Chemistry in the premedical curriculum: Viewpoint of the college of liberal arts. W. T. CALDWELL AND WILLIAM ROGERS, JR. 334
Chemistry, Invitation to: R.S.V.P. WILLIAM J. WISWESSER 271
Chemistry of benzene hexachloride and its insecticidal properties, The. GEORGE R. CHAMLIN 283
Chemistry of tooth tissues, The. RALPH W. PHILLIPS AND GRANT VAN HUYSEN 579
Chemistry, organic, Objective tests in 447
Chemistry, physical, Conversion factors in. PAUL PORTER SUTTON 126
Chemistry, plastics, A brief review of. ROBERT P. COURTNEY 306
Chemistry, Selecting a graduate school for training in. JOHN R. SAMPEY 583
Chemistry, The application of, to animal husbandry. J. G. ARCHIBALD 49
Chemist's curriculum, Russian in the. J. G. TOLPIN 123
Chemist's prayer, A. THERESE SOUTHGATE 507
Chemists, library, More wanted. ELSE L. SCHULZE 176
Classification of compounds, A new system for the: A contribution from the survey of antimalarial drugs. F. Y. WISELOGLE ET AL 375
Claude Louis Berthollet. PIERRE LEMAY AND RALPH E. OESPER 158, 230
Cleaning bath, Laboratory acid. GILBERT E. MOOS 465
Cobalt, nickel, zinc, and manganese, Separation and identification of. R. K. MCALPINE 298
Cobalt returns 522
Collector, A condensed water. H. W. STERNBERG AND B. T. FISHER 397
College chemistry beginning courses, The equilibrium concept in. NORMAN HACKERMAN 45
College, liberal arts, Viewpoint of the: The objectives of premedical education. W. B. KEIGHTON 322
College, medical, The viewpoint of the: Chemistry in the premedical curriculum. D. BAILEY CALVIN AND B. M. HENDRIX 326
College, medical, Viewpoint of the: Scientific and humanistic objectives in premedical education. H. E. SETTERFIELD 330
College of liberal arts, Viewpoint of the: Chemistry in the premedical curriculum. W. T. CALDWELL AND WILLIAM ROGERS, JR. 334
College, Silliman, of Yale University 342
Colloid chemistry, The application of, to the manufacture of inflated articles. LANGLEY W. ISOM 620
Colloidal metals in nonaqueous solvents by the Bredig method. ROBERT C. CROSSON, JR., AND HAROLD J. ABRAHAMS 289
Colorless to pink by adding acids, Changing phenolphthalein from. LEON MCCULLOCH 473
Column, fractionating, Construction of a. ROBERT TAFT, JR., AND CALVIN A. VANDERWERF 82
Combustion, Demonstration of, by catalysis. LEON MCCULLOCH 524
Committees and officers of the division of chemical education 48
Company, life insurance, Chemistry and a. SIDNEY H. ROBERTS 568
Complex ions, Inner: A chelate species. H. A. LIEBHAFSKY 341
Composition, Percentage, and the atomic theory. R. K. MCALPINE 227
Compounds, A new system for the classification of: A contribution from the survey of antimalarial drugs. F. Y. WISELOGLE ET AL 375
Compounds, Cationic and anionic: I, Inorganic nitrosyl compounds. THERALD MOELLER 441
Compounds, Coordination: II, Inorganic nitrosyl compounds. THERALD MOELLER 542
Compounds, Inorganic nitrosyl. THERALD MOELLER.
I. Cationic and anionic compounds 441
II. Coordination compounds 542
Compounds, organic, Patentability of. I. J. FELLNER 555
Concept, The equilibrium, in beginning college chemistry courses. NORMAN HACKERMAN 45
Condensation reactions: A laboratory preparation of ethylidene glycerol. M. MARTIN MAGLIO AND CHARLES A. BURGER 174
Condensed water collector, A. H. W. STERNBERG AND B. T. FISHER 397
Condenser-receiver unit, A. S. J. POTEMPA AND F. P. CASSARETTO 219
Conductivity demonstration, Use of the neon bulb for. G. W. THIESSEN 267
Conductivity method, The thermal, of gas analysis. CLARKE C. MINTER 237
Construction of a fractionating column. ROBERT TAFT, JR., AND CALVIN A. VANDERWERF 82
Contribution from the survey of antimalarial drugs, A: A new system for the classification of compounds. F. Y. WISELOGLE ET AL 375
Contributions to the history of chemistry, His: Max Abramovich Blokh. HERBERT S. KLICKSTEIN AND HENRY M. LEICESTER 451
Control, Conversion of a bomb-release, to activate a solenoid stirrer. JOHN F. SUTTLE, LYNNE L. MERRITT, JR., AND GEORGE F. SIDDONS 612
Control, Electrolytic, of corrosion. RONALD B. SPACHT 195
Conversion factors in physical chemistry. PAUL PORTER SUTTON 126
Conversion of a bomb-release control to activate a solenoid stirrer. JOHN F. SUTTLE, LYNNE L. MERRITT, JR., AND GEORGE F. SIDDONS 612
Coordination compounds: II, Inorganic nitrosyl compounds. THERALD MOELLER 542
Cornell, Summer courses in chemical microscopy at 194
Correspondence 46, 89, 155, 205, 246, 309, 348, 467, 516, 602
Corrosion, Electrolytic control of. RONALD B. SPACHT 195
Corrosion resistance of aluminum and its alloys, The. RONALD B. SPACHT 253
"Co-solvency" and Solvent action: A lecture demonstration. SANTI R. PALIT 182
Course, elementary chemistry, Catalysis and the. J. A. CAMPBELL 582
Course in quantitative analysis, A navy V-12. GEORGE W. BENNETT 247
Courses, beginning college chemistry, The equilibrium concept in. NORMAN HACKERMAN 45
Courses, Summer, in chemical microscopy at Cornell 194
Crystallization. CHARLES H. STONE 404
Cultural values of the sciences in the premedical program, The. W. B. MELDRUM AND W. E. CADBURY, JR. 338
Curriculum, premedical, Chemistry in the: The viewpoint of the medical college. D. BAILEY CALVIN AND B. M. HENDRIX 326
Curriculum, premedical, Chemistry in the: Viewpoint of the college of liberal arts. W. T. CALDWELL AND WILLIAM ROGERS, JR. 334
Curriculum, Russian in the chemist's. J. G. TOLPIN 123

DEMANDS, Peace, for aluminum expand. DAISY L. SCHOOLEY 399
Demonstration, A: Elastic silica. JAMES W. COLE 97
Demonstration, A lecture: Solvent action and "co-solvency." SANTI R. PALIT 182
Demonstration and Illustration in elementary volumetric analysis. C. C. MELOCHE AND R. J. CARNEY 198
Demonstration, conductivity, Use of the neon bulb for. G. W. THIESSEN 267
Demonstration mass spectrometer, A. W. H. EBERHARDT 220
Demonstration of combustion by catalysis. LEON MCCULLOCH 524
Demonstration of the existence of a free radical. L. MICHAELIS 317
Detection, The semimicro, of thiocyanate and halide ions. LYMAN E. PORTER 402
Determination, A Kjeldahl, of protein for general chemistry. EDWARD M. COLLINS AND ROBERT W. SHELTON 549
Development, photographic, Recent hypotheses concerning the mechanism of. T. H. JAMES 595
Device for teaching chemical arithmetic, A. HORACE G. DEMING 259
Devices, Simple, for measuring out small quantities of liquid. F. C. CALKINS 604
Dilute electrolytes, Sensitivity of precipitation reactions of. R. K. MCALPINE 28
Dioxide, Reactions of sulfur, with unsaturated hydrocarbons. OLIVER GRUMMITT AND ALAN ARDIS 73
Dipole moments, Measurement of. PAUL BENDER 179
Discoveries leading to a chain reaction: Uranium fission. MAXWELL L. EIDINOFF 60
Division of chemical education of the American Chemical Society 618
Division of chemical education, Officers and committees of the 48
Drugs, antimalarial, A contribution from the survey of: A new system for the classification of compounds. F. Y. WISELOGLE ET AL 375
Dryer, A simple laboratory hot-air. I. R. C. BICK 127

E.S.M.W.T., Wartime chemical education through. AUSTIN M. PATTERSON 463
Editor's Outlook (Editorials) 1, 53, 105, 157, 209, 261, 313, 365, 417, 469, 521, 573
Editors, Why, get gray 65
Education, chemical, Division of, of the American Chemical Society 618
Education, chemical, Officers and committees of the division of 48
Education, premedical, Scientific and humanistic objectives in: Viewpoint of the medical college. H. E. SETTERFIELD 330
Education, premedical, Symposium on 322–40
Education, premedical, The objectives of: Viewpoint of the liberal arts college. W. B. KEIGHTON 322
Education, Wartime chemical, through E.S.M.W.T. AUSTIN M. PATTERSON 463
Effective training in quantitative analysis. CHARLES B. DEWITT AND L. B. ROGERS 169
Elastic silica: A demonstration. JAMES W. COLE 97
Electrolytes, dilute, Sensitivity of precipitation reactions of. R. K. MCALPINE 28
Electrolytic control of corrosion. RONALD B. SPACHT 195
Electronic theories, Application of: Rearrangement of substituted benzopinacols. GUIDO H. STEMPEL, JR. 434

Electronics, improved, The basis of. DOUGLAS M. CONSIDINE AND DONALD P. ECKMAN 274
Electrons, Why do we believe in? ANTHONY STANDEN 356
Electropolishing process for silver 537
Elementary chemistry course, Catalysis and the. J. A. CAMPBELL 582
Elementary volumetric analysis, Illustration and demonstration in. C. C. MELOCHE AND R. J. CARNEY 198
Energetics, Nuclear. RUSSELL R. WILLIAMS, JR. 508
Engineer, radio, Faraday and the. R. H. WRIGHT 251
Entropy and the second law of thermodynamics. ARTHUR E. MARTELL 166
Equation of state, reduced, Note on van der Waal's. CHARLES A. STEVENSON 148
Equations, Balancing, for organic oxidation-reduction reactions. LLOYD N. FERGUSON 550
Equilibrium, Catalyzing the approach to. JOEL H. HILDEBRAND 589
Equilibrium concept in beginning college chemistry courses, The. NORMAN HACKERMAN 45
Errata: Chemical Russian, self-taught 248
Ethylidene glycerol, A laboratory preparation of: Condensation reactions. M. MARTIN MAGLIO AND CHARLES A. BURGER 174
Evaluation of structure, Refractometric analysis as a key to the: A system of characterization of pure hydrocarbons. RAY T. WENDLAND 3
Exchange, Ion. H. F. WALTON 454
Existence of a free radical, Demonstration of the. L. MICHAELIS 317
Exposition, The Lavoisier bicentenary, in Paris. H. S. VAN KLOOSTER 210

FACTORS, Conversion, in physical chemistry. PAUL PORTER SUTTON 126
Faraday and the radio engineer. R. H. WRIGHT 251
Faradays, Youthful. CHARLES H. STONE 249
Fichter, Friedrich. RALPH E. OESPER 394
Fission, Uranium: Discoveries leading to a chain reaction. MAXWELL L. EIDINOFF 60
Fluorochemistry of uranium, The. JACK DEMENT 213
Foreign technical publications. FLORENCE HARDEN 489
Form, An inventory, for a small laboratory. VELMA WILSON 165
Fractionating column, Construction of a. ROBERT TAFT, JR., AND CALVIN A. VANDERWERF 82
Fractionation of isotopes, The. A. M. SQUIRES 538
Franklin and Lavoisier. H. S. VAN KLOOSTER 107
Free radical, Demonstration of the existence of a. L. MICHAELIS 317
Friedrich Fichter. RALPH E. OESPER 394
Frontispieces:
BERTHOLLET, CLAUDE LOUIS (April)
BESSEMER CONVERTERS BLOWING FOR PEACE (Oct.)
CARVER, GEORGE WASHINGTON (June)
FRIEDEL, CHARLES (Jan.)
KARRER, PAUL (Aug.)
LEST WE FORGET (Feb.)
PAULZE, MARIE-ANNE-PIERRETTE (May)
PHILOSOPHER SHOWING AN EXPERIMENT ON THE AIR PUMP, A (Nov.)
PHOTOGRAPHIC MURAL, A (Sept.)
POGGENDORFF, JOHANN CHRISTIAN (March)
SILLIMAN, BENJAMIN (July)
WORLD'S LARGEST BUBBLE (Dec.)
Functions, thermodynamic, A presentation of the. C. C. COFFIN 584

GAS analysis, The thermal conductivity method of. CLARKE C. MINTER 237
Gasometric analysis, A: Analysis of sodium nitrite by sulfamic acid. ROBERT C. BRASTED 320
General chemistry, A Kjeldahl determination of protein for. EDWARD M. COLLINS AND ROBERT W. SHELTON 549
Genuine and synthetic: Rubies and sapphires. A. E. ALEXANDER 418
George Washington Carver: An American scientist. CLARENCE W. WRIGHT 268
Glass systems, Nonsilica. F. L. JONES 608
Glue, animal, The properties of. H. B. SWEATT 192
Glycerol, ethylidene, A laboratory preparation of: Condensation reactions. M. MARTIN MAGLIO AND CHARLES A. BURGER 174
Graduate assistants: Their stipends. ALEXANDER SILVERMAN 351
Graduate school, Selecting a, for training in chemistry. JOHN R. SAMPEY 583
Gray, Why editors get 65
Greek pigments, Ancient. EARLE R. CALEY 314
Group III, The qualitative analysis of: A survey. MARY MARGARET TENBUSCH AND GEORGE E. F. BREWER 66

HALIDE and thiocyanate ions, The semimicro detection of. LYMAN E. PORTER 402
Halides, nitrogen, New interest in the. FRED W. SCHUELER 168
Halogens, The positive character of the. JACOB KLEINBERG 559
Heats of solution, On. WILLIAM S. HORTON 393
Helium II, liquid, The anomalous behavior of. SIMON A. WEISSMAN 223
Here and There in the Trade Literature 37, 85, 140, 190, 244, 294, 345, 410, 562
Hexachloride, The chemistry of benzene, and its insecticidal properties. GEORGE R. CHAMLIN 283
History of chemistry, His contributions to the: Max Abramovich Blokh. HERBERT S. KLICKSTEIN AND HENRY M. LEICESTER 451
Hot-air dryer, A simple laboratory. I. R. C. BICK 127
Hot stage, A versatile microscope. WILLIAM A. BONNER 601
How to solve problems and influence students. THOMAS H. HAZLEHURST 445
Humanistic and Scientific objectives in premedical education: Viewpoint of the medical college. H. E. SETTERFIELD 330
Humanities, Scientific values of the, in the premedical program. DEWITT STETTEN, JR. 336
Husbandry, animal, The application of chemistry to. J. G. ARCHIBALD 49
Hydrocarbons, pure, A system of characterization of: Refractometric analysis as a key to the evaluation of structure. RAY T. WENDLAND 3
Hydrocarbons, unsaturated, Reactions of with sulfur dioxide. OLIVER GRUMMITT AND ALAN ARDIS 73
Hypotheses, Recent, concerning the mechanism of photographic development. T. H. JAMES 595

IDENTIFICATION and separation of cobalt, nickel, zinc, and manganese. R. K. MCALPINE 301
If I had a son. CYRIL E. ABBOTT 438
Illustration and demonstration in elementary volumetric analysis. C. C. MELOCHE AND R. J. CARNEY 198
Importance, The scientific, of the nuclear power projects. C. D. CORYELL 395
Improved electronics, The basis of. DOUGLAS M. CONSIDINE AND DONALD P. ECKMAN 274
Indexing, bibliography, and abstracting of chemical literature, A punched card method for the. CLOYD DAKE GULL 500
Indispensable leaf yellow. HAROLD H. STRAIN 262
Industrial organization, Operating an abstracting and information service within an. W. C. E. BARNES 152
Industrial uses of phosphates. PAUL LOGUE 529
Inflated articles, The application of colloid chemistry to the manufacture of. LANGLEY W. ISOM 620
Influence students, How to solve problems and. THOMAS H. HAZLEHURST 445
Influenza virus. MAX A. LAUFFER 530
Information and abstracting service within an industrial organization, Operating an. W. C. E. BARNES 152
Inner complex ions: A chelate species. H. A. LIEBHAFSKY 341
Inorganic nitrosyl compounds. THERALD MOELLER.
I. Cationic and anionic compounds 441
II. Coordination compounds 542
Inorganic Russian chemical nomenclature. III, Chemical Russian, self-taught. JAMES W. PERRY 116
Insecticidal properties, The chemistry of benzene hexachloride and its. GEORGE R. CHAMLIN 283
Instrumentation in chemical analysis. RAYMOND H. LAMBERT 610
Insurance company, life, Chemistry and a. SIDNEY H. ROBERTS 568
Interest, New, in the nitrogen halides. FRED W. SCHUELER 168
Introduction to thermodynamics, The. DUNCAN MACRAE 366
Inventory form for a small laboratory, An. VELMA WILSON 165
Invitation to chemistry: R.S.V.P. WILLIAM J. WISWESSER 271
Ion exchange. H. F. WALTON 454
Ions, Inner complex: A chelate species. H. A. LIEBHAFSKY 341
Ions, thiocyanate and halide, The semimicro detection of. LYMAN E. PORTER 402
Isotopes, The fractionation of. A. M. SQUIRES 538

KARRER, PAUL. RALPH E. OESPER 392
Key to the evaluation of structure, Refractometric analysis as a: A system of characterization of pure hydrocarbons. RAY T. WENDLAND 3
Kjeldahl determination of protein for general chemistry, A. EDWARD M. COLLINS AND ROBERT W. SHELTON 549
Knowledge, Chemical, in the New Testament. HUGO ZAHND AND DOROTHY GILLIS 90, 128

LABORATORY, A simple, hot-air dryer. I. R. C. BICK 127
Laboratory acid cleaning bath. GILBERT E. MOOS 465
Laboratory preparation of ethylidene glycerol, A: Condensation reactions. M. MARTIN MAGLIO AND CHARLES A. BURGER 174
Laboratory, small, An inventory form for a. VELMA WILSON 165
Lavoiser and Franklin. H. S. VAN KLOOSTER 107
Lavoisier bicentenary exposition in Paris, The. H. S. VAN KLOOSTER 210
Law of thermodynamics, second, Entropy and the. ARTHUR E. MARTELL 166
Leaf pigments. SAMUEL E. KAMERLING 202
Leaf yellow, Indispensable. HAROLD H. STRAIN 262
Lecture demonstration, A: Solvent action and "co-solvency." SANTI R. PALIT 182
Let your librarian look it up. RAYMOND R. DICKISON 175
Liberal arts college, Viewpoint of the: The objectives of premedical education. W. B. KEIGHTON 322
Liberal arts, Viewpoint of the college of: Chemistry in the premedical curriculum. W. T. CALDWELL AND WILLIAM ROGERS, JR. 334
Librarian, Let your, look it up. RAYMOND R. DICKISON 175
Library chemists, More, wanted. ELSE L. SCHULZE 176
Library techniques, technical, Its relation to: The American patent system. CASPER W. OOMS 478
Library techniques, technical, Symposium number three on 474–507
Library, The technical, of tomorrow. NORMAN C. HILL 474
Life insurance company, Chemistry and a. SIDNEY H. ROBERTS 568
Light and active 444
Liquid helium II, The anomalous behavior of. SIMON A. WEISSMAN 223
Liquid, Simple devices for measuring out small quantities of. F. C. CALKINS 604
Literature, chemical, A punched card method for the bibliography, abstracting, and indexing of. CLOYD DAKE GULL 500
Literature, Russian technical, Searching the. J. G. TOLPIN 485
Low-ester pectins in ready-to-serve salads. 459

MAGNETIC anisotropy of organic solids. P. W. SELWOOD AND JOHN PARODI 574
Manganese, cobalt, zinc, and nickel, Separation and identification of. R. K. MCALPINE 298
Manufacture of inflated articles, The application of colloid chemistry to the. LANGLEY W. ISOM 620
Marcano, Vicente (1848–1891): A pioneer chemist of Venezuela. Conrado F. Asenjo 145
Mass spectrometer, A demonstration. W. H. EBERHARDT 220
Max Abramovich Blokh: His contributions to the history of chemistry. HERBERT S. KLICKSTEIN AND HENRY M. LEICESTER 451
Measurement of dipole moments. PAUL BENDER 179
Measuring out small quantities of liquid, Simple devices for. F. C. CALKINS 604
Mechanism of photographic development, Recent hypotheses concerning the. T. H. JAMES 595
Medical college, The viewpoint of the: Chemistry in the premedical curriculum. D. BAILEY CALVIN AND B. M. HENDRIX 326
Medical college, Viewpoint of the: Scientific and humanistic objectives in premedical education. H. E. SETTERFIELD 330
Medicinals, synthetic, A survey of. C. PAULINE BURT 358, 412

Metals, Colloidal, in nonaqueous solvents by the Bredig method. ROBERT C. CROSSON, JR., AND HAROLD J. ABRAHAMS.... 289
Metals, Nonferrous, aid in steel's use. JACOB LEVIN.... 142
Methionine.... 548
Method, A punched card, for the bibliography, abstracting, and indexing of chemical literature. CLOYD DAKE GULL.... 500
Method, Bredig, Colloidal metals in nonaqueous solvents by the. ROBERT C. CROSSON, JR., AND HAROLD J. ABRAHAMS.... 289
Method, The thermal conductivity, of gas analysis. CLARKE C. MINTER.... 237
Methods of separation and tests for purity. HAROLD G. CASSIDY.... 427
Methods, Statistical, of research. HUGH M. SMALLWOOD.... 352
Metric system, Note on teaching the. G. WAKEHAM.... 134
Microscope hot stage, A versatile. WILLIAM A. BONNER.... 601
Microscopy, chemical, Summer courses in, at Cornell.... 194
Moments, dipole, Measurement of. PAUL BENDER.... 179
More library chemists wanted. ELSE L. SCHULZE.... 176

New England Association of Chemistry Teachers:
Articles
Activated carbon. W. A. HELBIG.... 98
Application of chemistry to animal husbandry. J. G. ARCHIBALD.... 49
Application of colloid chemistry to the manufacture of inflated articles, The. LANGLEY W. ISOM.... 620
Brief review of plastics chemistry, A. ROBERT P. COURTNEY.... 306
Chemistry and a life insurance company. SIDNEY H. ROBERTS.... 568
Leaf pigments. SAMUEL E. KAMERLING 202
Survey of synthetic medicinals, A. C. PAULINE BURT.... 358, 412
Demonstrations.... 154
Eighth summer conference.... 258, 466
Report of the NEACT.... 49, 98, 154, 202, 258, 306, 358, 412, 466, 518, 568, 620
Navy V-12 course in quantitative analysis, A. GEORGE W. BENNETT.... 247
Neon bulb, Use of the, for conductivity demonstration. G. W. THIESSEN.... 267
New interest in the nitrogen halides. FRED W. SCHUELER.... 168
New system for the classification of compounds, A: A contribution from the survey of antimalarial drugs. EMMETT L. BUHLE, ELINOR D. HARTNELL, ALEXANDER M. MOORE, LOUISE R. WISELOGLE, AND F. Y. WISELOGLE.... 375
New testament, Chemical knowledge in the. HUGO ZAHND AND DOROTHY GILLIS.... 90, 128
Nickel, cobalt, zinc, and manganese, Separation and identification of. R. K. MCALPINE.... 298
Nitrite, sodium, Analysis of by sulfamic acid: A gasometric analysis. ROBERT C. BRASTED.... 320
Nitrites. CHARLES H. STONE.... 196
Nitrogen halides, New interest in the. FRED W. SCHUELER.... 168
Nitrosyl compounds, Inorganic. THERALD MOELLER.
I. Cationic and anionic compounds.... 441
II. Coordination compounds.... 542
No longer pointless.... 2
Nomenclature, Russian inorganic chemical. III. Chemical Russian, self-taught. JAMES W. PERRY.... 116
Nonaqueous solvents, Colloidal metals in, by the Bredig method. ROBERT C. CROSSON, JR., AND HAROLD J. ABRAHAMS.... 289
Nonferrous metals aid in steel's use. JACOB LEVIN.... 142
Nonsilica glass systems. F. L. JONES.... 608
Note on teaching the metric system. G. WAKEHAM.... 134
Note on van der Waal's reduced equation of state. CHARLES A. STEVENSON.... 148
Notice to authors.... 636
Nuclear energetics. RUSSELL R. WILLIAMS, JR.... 508
Nuclear power projects, The scientific importance of the. C. D. CORYELL.... 395
Nuclear reactions, RUSSELL R. WILLIAMS, JR.... 423

OBJECTIVE tests in organic chemistry.... 447
Objectives of premedical education, The: Viewpoint of the liberal arts college. W. B. KEIGHTON.... 322
Objectives of qualitative analysis, The. W. L. WASLEY.... 357
Objectives, scientific and humanistic, in premedical education: Viewpoint of the medical college. H. E. SETTERFIELD.... 330
Odor and the organic chemist. EVERETT L. SAUL.... 296
Officers and committees of the division of chemical education.... 48
On heats of solution. WILLIAM S. HORTON. 393
Operating an abstracting and information service within an industrial organization. W. C. E. BARNES.... 152
Organic chemist, Odor and the. EVERETT L. SAUL.... 296
Organic chemistry, Objective tests in.... 447
Organic compounds, Patentability of. I. J. FELLNER.... 555
Organic oxidation-reduction reactions, Balancing equations for. LLOYD N. FERGUSON.... 550
Organic preparations, Some small-scale. L. O. BINDER.... 285
Organic solids, Magnetic anisotropy of. P. W. SELWOOD AND JOHN PARODI.... 574
Organics, Silicon. HAROLD HAUSMAN.... 16
Organization, industrial, Operating an abstracting and information service within an. W. C. E. BARNES.... 152
Organization of the technical report. FLOYD O. SLATE.... 439
Out of the Editor's Basket.... 39, 86, 136, 186, 240, 290, 346, 406, 460, 512, 564, 614
Oxidation-reduction reactions, organic, Balancing equations for. LLOYD N. FERGUSON.... 550
Oxidizing agents, Anomalous behavior of some. R. K. MCALPINE.... 301

PARIS, The Lavoisier bicentenary exposition in. H. S. VAN KLOOSTER.... 210
Particle size determination. ARTHUR A. VERNON.... 448
Patent system, The American: Its relation to technical library techniques. CASPER W. OOMS.... 478
Patentability of organic compounds. I. J. FELLNER.... 555
Patents, basic, Plants hold the. EDMUND W. SINNOTT.... 35
Paul Karrer. RALPH E. OESPER.... 392
Peace demands for aluminum expand. DAISY L. SCHOOLEY.... 399
Pectins, Low-ester, in ready-to-serve salads. 459
Percentage composition and the atomic theory. R. K. MCALPINE.... 227
Periodic table, Atomic size and the. J. A. CAMPBELL.... 525
Periodicals, Wartime scientific. E. J. CRANE.... 482
Phase rule and topology, The. IRVIN LEVIN.... 183
Phenolphthalein, Changing, from colorless to pink by adding acids. LEON MCCULLOCH.... 473
Phosphates, Industrial uses of. PAUL LOGUE.... 529
Photographic development, Recent hypotheses concerning the mechanism of. T. H. JAMES.... 595
Photography as a basic science. W. C. OELKE.... 593
Physical chemistry, Conversion factors in. PAUL PORTER SUTTON.... 126
Pigments, Ancient Greek. EARLE R. CALEY.... 314
Pigments, Leaf. SAMUEL E. KAMERLING.... 202
Pink, Changing phenolphthalein from colorless to, by adding acids. LEON MCCULLOCH.... 473
Pioneer chemist of Venezuela, A: Vicente Marcano (1848–1891). CONRADO F. ASENJO.... 145
Plant alchemy.... 44
Plants hold the basic patents. EDMUND W. SINNOTT.... 35
Plastics chemistry, A brief review of. ROBERT P. COURTNEY.... 306
Pointless, No longer.... 2
Positive character of the halogens, The. JACOB KLEINBERG.... 559
Power projects, nuclear, The scientific importance of the. C. D. CORYELL.... 395
Prayer, A chemist's. THERESE SOUTHGATE.... 507
Precious sweep stakes, The.... 287
Precipitation reactions, Sensitivity of, of dilute electrolytes. R. K. MCALPINE. 28
Premedical curriculum, Chemistry in the: The viewpoint of the medical college. D. BAILEY CALVIN AND B. M. HENDRIX 326
Premedical curriculum, Chemistry in the: Viewpoint of the college of liberal arts. W. T. CALDWELL AND WILLIAM ROGERS, JR.... 334
Premedical education, Scientific and humanistic objectives in: Viewpoint of the medical college. H. E. SETTERFIELD.... 330
Premedical education, Symposium on.... 322–40
Premedical education, The objectives of: Viewpoint of the liberal arts college. W. B. KEIGHTON.... 322
Premedical program, Scientific values of the humanities in the. DEWITT STETTEN, JR.... 336
Premedical program, The cultural values of the sciences in the. W. B. MELDRUM AND W. E. CADBURY, JR.... 338
Premedical students, Quantitative analysis for. ELTON M. BAKER.... 567
Preparation, A laboratory, of ethylidene glycerol: Condensation reactions. M. MARTIN MAGLIO AND CHARLES A. BURGER.... 174
Preparation of tellurium tetrachloride, An apparatus for the. JOHN F. SUTTLE AND ROBERT P. GECKLER.... 135
Preparations, Some small-scale organic. L. O. BINDER.... 285
Presentation of the thermodynamic functions, A. C. C. COFFIN.... 584
Problem, The vocabulary: II, Chemical Russian, self-taught. JAMES W. PERRY 22
Problems, How to solve, and influence students. THOMAS H. HAZLEHURST.. 445
Process, Electropolishing, for silver.... 537
Program, premedical, Scientific values of the humanities in the. DEWITT STETTEN, JR.... 336
Program, premedical, The cultural values of the sciences in the. W. B. MELDRUM AND W. E. CADBURY, JR.... 338
Projects, nuclear power, The scientific importance of the. C. D. CORYELL.. 395
Properties, insecticidal, The chemistry of benzene hexachloride and its. GEORGE R. CHAMLIN.... 283
Properties of animal glue, The. H. B. SWEATT.... 192
Protein, A Kjeldahl determination of, for general chemistry. EDWARD M. COLLINS AND ROBERT W. SHELTON... 549
Publications, Foreign technical. FLORENCE HARDEN.... 489
Punch card techniques and applications. R. S. CASEY, C. F. BAILEY, AND G. J. COX.... 495
Punched card method for the bibliography, abstracting, and indexing of chemical literature, A. CLOYD DAKE GULL.... 500
Pure hydrocarbons, A system of characterization of: Refractometric analysis as a key to the evaluation of structure. RAY T. WENDLAND.... 3
Purity, tests for, and Methods of separation. HAROLD G. CASSIDY.... 427

QUALITATIVE analysis of group III, The: A survey. MARY MARGARET TENBUSCH AND GEORGE E. F. BREWER 66
Qualitative analysis, The objectives of. W. L. WASLEY.... 357
Quantitative analysis, A navy V-12 course in. GEORGE W. BENNETT.... 247
Quantitative analysis, Effective training in. CHARLES B. DEWITT AND L. B. ROGERS.... 169
Quantitative analysis for premedical students. ELTON M. BAKER.... 567
Quantities of liquid, small, Simple devices for measuring out. F. C. CALKINS... 604

RSVP.: Invitation to chemistry. WILLIAM J. WISWESSER.... 271
Radical, free, Demonstration of the existence of a. L. MICHAELIS.... 317
Radio engineer and Faraday. R. H. WRIGHT.... 251
Reaction, chain, Discoveries leading to a: Uranium fission. MAXWELL L. EIDINOFF.... 60
Reactions, Condensation: A laboratory preparation of ethylidene glycerol. M. MARTIN MAGLIO AND CHARLES A. BURGER.... 174
Reactions from our readers.... 405
Reactions, Nuclear. RUSSELL R. WILLIAMS, JR.... 423
Reactions of sulfur dioxide with unsaturated hydrocarbons. OLIVER GRUMMITT AND ALAN ARDIS.... 73
Reactions, organic oxidation-reduction, Balancing equations for. LLOYD N. FERGUSON.... 550
Reactions, Sensitivity of precipitation, of dilute electrolytes. R. K. MCALPINE. 28
Readers, Reactions from our.... 405
Ready-to-serve salads, Low-ester pectins in.... 459
Rearrangement of substituted benzopinacols: Application of electronic theories. GUIDO H. STEMPEL, JR.... 434
Receiver-condenser unit, A. S. J. POTEMPA AND F. P. CASSARETTO.... 219
Recent Books.... 52, 103, 155, 206, 260, 310, 362, 416, 468, 520, 572, 624

Recent hypotheses concerning the mechanism of photographic development. T. H. JAMES ... 595
Reduced equation of state, Note on van der Waal's. CHARLES A. STEVENSON ... 148
Reduction-oxidation reactions, organic, Balancing equations for. LLOYD N. FERGUSON ... 550
Refractometric analysis as a key to the evaluation of structure: A system of characterization of pure hydrocarbons. RAY T. WENDLAND ... 3
Relation to technical library techniques, Its: The American patent system. CASPER W. OOMS ... 478
Report, technical, Organization of the. FLOYD O. SLATE ... 439
Report writing, technical, Training for. FLOYD O. SLATE ... 398
Research, Statistical methods of. HUGH M. SMALLWOOD ... 352
Resistance, The corrosion, of aluminum and its alloys. RONALD B. SPACHT ... 253
Review, A brief, of plastics chemistry. ROBERT P. COURTNEY ... 306
Rubies and sapphires: Genuine and synthetic. A. E. ALEXANDER ... 418
Rule, The phase, and topology. IRVIN LEVIN ... 183
Russian, Chemical, self-taught. JAMES W. PERRY.
II. The vocabulary problem ... 22
III. Russian inorganic chemical nomenclature ... 116
Errata ... 248
Russian in the chemist's curriculum. J. G. TOLPIN ... 123
Russian inorganic chemical nomenclature. III. Chemical Russian, self-taught. JAMES W. PERRY ... 116
Russian technical literature, Searching the. J. G. TOLPIN ... 485

SALADS, ready-to-serve, Low-ester pectins in ... 459
Salt shaker wedding. ELIZABETH THOMPSON ... 350
Sapphires and Rubies: Genuine and synthetic. A. E. ALEXANDER ... 418
School, graduate, Selecting a, for training in chemistry. JOHN R. SAMPEY ... 583
Science, basic, Photography as a. W. C. OELKE ... 593
Science seminars. SISTER M. IGNATIA, I.H.M. ... 149
Science teachers and the atomic bomb. RICHARD M. NOYES ... 343
Sciences in the premedical program, The cultural values of the. W. B. MELDRUM AND W. E. CADBURY, JR. ... 338
Scientific and humanistic objectives in premedical education: Viewpoint of the medical college. H. E. SETTERFIELD ... 330
Scientific importance of the nuclear power projects, The. C. D. CORYELL ... 395
Scientific periodicals, Wartime. E. J. CRANE ... 482
Scientific values of the humanities in the premedical program. DEWITT STETTEN, JR. ... 336
Scientist, An American: George Washington Carver. CLARENCE W. WRIGHT ... 268
Searching the Russian technical literature. J. G. TOLPIN ... 485
Second law of thermodynamics, Entropy and the. ARTHUR E. MARTELL ... 166
Selecting a graduate school for training in chemistry. JOHN R. SAMPEY ... 583
Selenium. CHARLES H. STONE ... 349
Self-taught Chemical Russian. JAMES W. PERRY.
II. The vocabulary problem ... 22
III. Russian inorganic chemical nomenclature ... 116
Errata ... 248
Semantics of thermodynamics, The. W. F. LUDER.
Part I ... 54
Part II ... 110
Semimicro detection of thiocyanate and halide ions, The. LYMAN E. PORTER ... 402
Seminars, Science. SISTER M. IGNATIA, I.H.M. ... 149
Sensitivity of precipitation reactions of dilute electrolytes. R. K. MCALPINE ... 28
Separation and identification of cobalt, nickel, zinc, and manganese. R. K. McAlpine ... 298
Separation, Methods of, and tests for purity. HAROLD G. CASSIDY ... 427
Service, Operating an abstracting and information, within an industrial organization. W. C. E. BARNES ... 152
Silica, Elastic: A demonstration. JAMES W. COLE ... 97
Silicon organics. HAROLD HAUSMAN ... 16
Silliman college of Yale University ... 342
Silver, Electropolishing process for ... 537
Simple devices for measuring out small quantities of liquid. F. C. CALKINS ... 604
Simple laboratory hot-air dryer, A. I. R. C. BICK ... 127
Simple sublimation apparatus, A. JOHN PITHA ... 403
Size, Atomic, and the periodic table. J. A. CAMPBELL ... 525
Small laboratory, An inventory form for a. VELMA WILSON ... 165
Small quantities of liquid, Simple devices for measuring out. F. C. CALKINS ... 604
Small-scale organic preparations, Some. L. O. BINDER ... 285
Society, American Chemical, Division of chemical education of the ... 618
Sodium nitrite by sulfamic acid, Analysis of: A gasometric analysis. ROBERT C. BRASTED ... 320
Solenoid stirrer, Conversion of a bomb-release control to activate a. JOHN F. SUTTLE, LYNNE L. MERRITT, JR., AND GEORGE F. SIDDONS ... 612
Solids, organic, Magnetic anisotropy of. P. W. SELWOOD AND JOHN PARODI ... 574
Solution, On heats of. WILLIAM S. HORTON ... 393
Solve problems and influence students, How to. THOMAS H. HAZLEHURST ... 445
Solvent action and "co-solvency": A lecture demonstration. SANTI R. PALIT ... 182
Solvents, Colloidal metals in nonaqueous, by the Bredig method. ROBERT C. CROSSON, JR., AND HAROLD J. ABRAHAMS ... 289
Some small-scale organic preparations. L. O. BINDER ... 285
Son, If I had a. CYRIL E. ABBOTT ... 438
Species, A chelate: Inner complex ions. H. A. LIEBHAFSKY ... 341
Spectrometer, A demonstration mass. W. H. EBERHARDT ... 220
Stage, A versatile microscope hot. WILLIAM A. BONNER ... 601
Stakes, The precious sweep ... 287
State, Note on van der Waal's reduced equation of. CHARLES A. STEVENSON ... 148
Statistical methods of research. HUGH M. SMALLWOOD ... 352
Steel technology, Wartime advances in ... 470
Steel's use, Nonferrous metals aid in. JACOB LEVIN ... 142
Stipends, Their: Graduate assistants. ALEXANDER SILVERMAN ... 351
Stirrer, solenoid, Conversion of a bomb-release control to activate a. JOHN F. SUTTLE, LYNNE L. MERRITT, AND GEORGE F. SIDDONS ... 612
Structure, Refractometric analysis as a key to the evaluation of: A system of characterization of pure hydrocarbons. RAY T. WENDLAND ... 3
Students, How to solve problems and influence. THOMAS H. HAZLEHURST ... 445
Students, premedical, Quantitative analysis for. ELTON M. BAKER ... 567
Sublimation apparatus, A simple. JOHN PITHA ... 403
Substituted benzopinacols, Rearrangement of: Application of electronic theories. GUIDO H. STEMPEL, JR. ... 434
Sulfamic acid, Analysis of sodium nitrite by: A gasometric analysis. ROBERT C. BRASTED ... 320
Sulfur dioxide, Reactions of, with unsaturated hydrocarbons. OLIVER GRUMMITT AND ALAN ARDIS ... 73
Summer courses in chemical microscopy at Cornell ... 194
Survey, A: The qualitative analysis of group III. MARY MARGARET TENBUSCH AND GEORGE E. F. BREWER ... 66
Survey of antimalarial drugs, A contribution from the: A new system for the classification of compounds. F. Y. WISELOGLE ET AL ... 375
Survey of synthetic medicinals, A. C. PAULINE BURT ... 358, 412
Sweep stakes, The precious ... 287
Symposium number three on technical library techniques ... 474–507
Symposium on premedical education ... 322–40
Synthetic and Genuine: Rubies and sapphires. A. E. ALEXANDER ... 418
Synthetic medicinals, A survey of. C. PAULINE BURT ... 358, 412
System, A new, for the classification of compounds: A contribution from the survey of antimalarial drugs. F. Y. WISELOGLE ET AL ... 375
System, metric, Note on teaching the. G. WAKEHAM ... 134
System of characterization of pure hydrocarbons, A: Refractometric analysis as a key to the evaluation of structure. RAY T. WENDLAND ... 3
System, The American patent: Its relation to technical library techniques. CASPER W. OOMS ... 478
Systems, Nonsilica glass. F. L. JONES ... 608

TABLE, periodic, Atomic size and the. J. A. CAMPBELL ... 525
Teachers, Science, and the atomic bomb. RICHARD M. NOYES ... 343
Teaching chemical arithmetic, A device for. HORACE G. DEMING ... 259
Teaching the metric system, Note on. G. WAKEHAM ... 134
Teaching total acidity. SISTER MARIE CLOTILDE ... 433
Teaching valence in beginning chemistry. G. WAKEHAM ... 43
Technical library of tomorrow, The. NORMAN C. HILL ... 474
Technical library techniques, Its relation to: The American Patent system. CASPER W. OOMS ... 478
Technical library techniques, Symposium number three on ... 474–507
Technical literature, Russian, Searching the. J. G. TOLPIN ... 485
Technical publications, Foreign. FLORENCE HARDEN ... 489
Technical report, Organization of the. FLOYD O. SLATE ... 439
Technical report writing, Training for. FLOYD O. SLATE ... 398
Techniques, Punch card, and applications. R. S. CASEY, C. F. BAILEY, AND G. J. COX ... 495
Techniques, technical library, Its relation to: The American patent system. CASPER W. OOMS ... 478
Techniques, technical library, Symposium number three on ... 474–507
Technology, steel, Wartime advances in ... 470
Tellurium tetrachloride, An apparatus for the preparation of. JOHN F. SUTTLE AND ROBERT P. GECKLER ... 135
Testament, New, Chemical knowledge in the. HUGO ZAHND AND DOROTHY GILLIS ... 90, 128
Tests for purity and Methods of separation. HAROLD G. CASSIDY ... 427
Tests, Objective, in organic chemistry ... 447
Tetrachloride, tellurium, An apparatus for the preparation of. JOHN F. SUTTLE AND ROBERT P. GECKLER ... 135
Their stipends: Graduate assistants. ALEXANDER SILVERMAN ... 351
Theories, electronic, Application of: Rearrangement of substituted benzopinacols. GUIDO H. STEMPEL, JR. ... 434
Theory, atomic, Percentage composition and the. R. K. MCALPINE ... 227
Thermal conductivity method of gas analysis, The. CLARKE C. MINTER ... 237
Thermodynamic functions, A presentation of the. C. C. COFFIN ... 584
Thermodynamics, Entropy and the second law of. ARTHUR E. MARTELL ... 166
Thermodynamics, The introduction to. DUNCAN MACRAE ... 366
Thermodynamics, The semantics of. W. F. LUDER.
Part I ... 54
Part II ... 110
Thiocyanate and halide ions, The semimicro detection of. LYMAN E. PORTER ... 402
Three, Symposium number, on technical library techniques ... 474–507
Tissues, tooth, The chemistry of. RALPH W. PHILLIPS AND GRANT VAN HUYSEN ... 579
Tomorrow, The technical library of. NORMAN C. HILL ... 474
Tooth tissues, The chemistry of. RALPH W. PHILLIPS AND GRANT VAN HUYSEN ... 579
Topic, An airy ... 205
Topology and The phase rule. IRVIN LEVIN ... 183
Total acidity, Teaching. SISTER MARIE CLOTILDE ... 433
Training, Effective, in quantitative analysis. CHARLES B. DEWITT AND L. B. ROGERS ... 169
Training for technical report writing. FLOYD O. SLATE ... 398
Training in chemistry, Selecting a graduate school for. JOHN R. SAMPEY ... 583

UNIT, A condenser-receiver. S. J. POTEMPA AND F. P. CASSARETTO ... 219
University, Yale, Silliman College of ... 342
Unsaturated hydrocarbons, Reactions of sulfur dioxide with. OLIVER GRUMMITT AND ALAN ARDIS ... 73
Uranium fission: Discoveries leading to a chain reaction. MAXWELL L. EIDINOFF ... 60
Uranium, The fluorochemistry of. JACK DEMENT ... 213
Use of the neon bulb for conductivity demonstration. G. W. THIESSEN ... 267

Use, steel's, Nonferrous metals aid in. Jacob Levin 142
Uses, Industrial, of phosphates. PAUL LOGUE 529

V-12 course, A navy, in quantitative analysis. GEORGE W. BENNETT 247
Valence, Teaching, in beginning chemistry. G. WAKEHAM 43
Values, Scientific, of the humanities in the premedical program. DEWITT STETTEN, JR. 336
Values, The cultural, of the sciences in the premedical program. W. B. MELDRUM AND W. E. CADBURY, JR. 338
Van der Waal's reduced equation of state, Note on. CHARLES A. STEVENSON 148
Venezuela, A pioneer chemist of: Vicente Marcano (1848–1891). CONRADO F. ASENJO 145
Versatile microscope hot stage, A. WILLIAM A. BONNER 601
Vicente Marcano (1848–1891): A pioneer chemist of Venezuela. CONRADO F. ASENJO 145
Viewpoint of the college of liberal arts: Chemistry in the premedical curriculum. W. T. CALDWELL AND WILLIAM ROGERS, JR. 334
Viewpoint of the liberal arts college: The objectives of premedical education. W. B. KEIGHTON 322
Viewpoint of the medical college: Scientific and humanistic objectives in premedical education. H. E. SETTERFIELD 330
Viewpoint of the medical college, The: Chemistry in the premedical curriculum. D. BAILEY CALVIN AND B. M. HENDRIX 326
Vocabulary problem, The: II, Chemical Russian, self-taught. JAMES W. PERRY 22
Volumetric analysis, elementary, Illustration and demonstration in. C. C. MELOCHE AND R. J. CARNEY 198

WANTED: More library chemists. ELSE L. SCHULZE 176
War victims—Books 312
Wartime advances in steel technology 470
Wartime chemical education through E.S.M.W.T. AUSTIN M. PATTERSON .. 463
Wartime scientific periodicals. E. J. CRANE 482
Water collector, A condensed. H. W. STERNBERG AND B. T. FISHER 397
Wedding, Salt shaker. ELIZABETH THOMPSON 350
What's Been Going On 106, 206
Why do we believe in electrons? ANTHONY STANDEN 356
Why editors get gray 65
Writing, technical report, Training for. FLOYD O. SLATE 398

YALE University, Silliman College of 342
Yellow, Indispensable leaf. HAROLD H. STRAIN 262
Youthful Faradays. CHARLES H. STONE .. 249

ZINC, cobalt, nickel, and manganese, Separation and identification of. R. K. MCALPINE 298

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